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TECHNICAL NOTE 3259

INVESTIGATION OF NICKEL-ALUMINUM ALLOYS CONTAINING
FROM 14 TO 34 PERCENT ALUMINUM

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INVESTIGATION OF NICKEL-ALUMINUM ALLOYS CONTAINING FROM

14 TO 34 PERCENT ALUMINUM

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SUMMARY

As part of a study of the properties of intermetallics, an investigation was made of nickel-aluminum alloys which were from 14 to 34 percent aluminum by weight. It is in this range that the Ni_3Al and NiAl intermetallics are found. These alloys were prepared by casting. As the aluminum content was increased, more difficulty was encountered in the preparation of sound, nonporous castings, apparently because of the increasingly exothermic nature of the reaction between the nickel and the aluminum. However, except for the alloys containing 34 percent aluminum and the stoichiometric NiAl (31.5 percent Al), all castings were sound and free from excessive porosity.

The Ni_3Al intermetallic compound had a room-temperature tensile strength of 48,450 pounds per square inch with 2.5- to 6.3-percent elongation. The NiAl cast alloys whose strength properties were determined ranged in composition from 25 to 30 percent aluminum. The maximum room-temperature tensile strength was 24,100 pounds per square inch for the 25-percent-aluminum alloy.

A mixture of phases Ni_3Al and NiAl occurred in the 17.5-percent-aluminum alloy. Of those investigated, this alloy had the most outstanding properties and was studied in greatest detail. The strength of the as-cast 17.5-percent-aluminum alloy at room temperature was 79,600 pounds per square inch with 1.2-percent elongation. At 1500° F, the tensile strength was 50,000 pounds per square inch. The 17.5-percent-aluminum alloy was readily rollable at 2400° F, resisted thermal shock, possessed outstanding oxidation resistance, and had a moderate impact strength. In creep-rupture at 1350° F, the 100-hour strength was 14,000 pounds per square inch. Compared with conventional high-temperature alloys, creep rates for this alloy were high. The effect of thermal treatment on microstructure was determined. It was found that a martensite-like transformation takes place during cooling at temperatures of 2200° F and above. The martensite-like transformation may be very useful for precipitation-hardening of the alloy.

INTRODUCTION

Intermetallics are known to possess properties radically different from those of their constituent elements. Also, many are stable at high temperatures. An example is the nickel-aluminum intermetallic NiAl, which has a high melting point compared with those of its constituent elements. Reference 1 reports good modulus-of-rupture strengths at temperatures up to 1800° F, and excellent oxidation resistance at temperatures up to 2000° F for the NiAl composition prepared by powder metallurgy. Reference 2 reports attempts to prepare NiAl by casting. The bodies proved to be extremely fragile and weak.

Several promising nickel-base alloys containing from 6 to 8 percent aluminum have recently been investigated (refs. 2 to 4). One such alloy (Guy's alloy (ref. 4)) has a 100-hour stress-rupture strength of 50,000 pounds per square inch at 1500° F. On a strength basis, this alloy would be satisfactory for turbine blades at operating temperatures of 1500° F and above. Since the aluminum in these alloys is to a large degree responsible for their oxidation resistance and contributes towards their high-temperature strength, a study was made of nickel-aluminum alloys containing higher percentages (14 to 34 percent) of aluminum.

The purpose of this investigation is to provide an exploratory study of the properties of nickel-aluminum alloys in the range 14 to 34 percent by weight aluminum. The Ni₃Al and NiAl intermetallics and mixtures of these intermetallics exist in this range (fig. 1). The room- and elevated-temperature strengths and ductility of these phases were of particular interest.

The alloys were prepared by casting, since this method readily lends itself to the production of sound bodies and complex shapes. Methods of melting and casting were therefore developed, and a series of nickel alloys with 14 to 34 percent aluminum was prepared. Tensile tests at room temperature and 1500° F were made, and elongations determined. Susceptibility to hot-rolling was investigated on another set of specimens of the same compositions. The 17.5-percent-aluminum alloy, which had the most promising properties with respect to both room-temperature tensile strength and hot formability, was selected for further investigation. This included studies of the creep-rupture strength at 1350° F, impact resistance, thermal shock behavior, oxidation resistance, and effects of thermal treatment on microstructure.

MATERIALS, APPARATUS, AND PROCEDURE

Specimen Preparation

Materials. - The chemical analyses of the raw materials used throughout the investigation are given in table I. The electrolytic

nickel was in the form of chips, and the 2S aluminum, used for most of the specimens, was charged as pieces of 1-inch-diameter bar stock. Several castings were prepared with high-purity aluminum to determine the difference, if any, between these specimens and those made with 2S aluminum. No improvement in tensile-strength properties was noted, and, therefore, all subsequent castings were produced from the less expensive and more available 2S stock.

Alloy compositions. - Comparison of the intended compositions and the actual compositions for typical castings are shown in table II. Table III lists the phases present in the compositions studied.

During the course of the investigation, small quantities of various grain-refining agents were added to the 17.5-percent-aluminum alloy to improve the as-cast grain structure. A series of castings were made containing boron, titanium, and molybdenum in quantities of 0.05, 0.3, and 0.5 percent, respectively. These were added in the form of granules of ferro-boron, titanium sponge, and molybdenum sheet.

Casting

All castings were prepared in the bell-jar vacuum-induction furnace shown in figure 2. The weight of the castings varied from 400 to 600 grams. After charging the alumina crucible, a vacuum of 2 to 10 microns was drawn, and tank argon was then introduced to a slightly positive pressure. The flat nickel chips at the bottom of the crucible acted as a susceptor in the induction field and raised the temperature of the aluminum to the melting point. The reaction between the molten aluminum and the nickel was violently exothermic, especially for the high-aluminum alloys, and the charge became molten in a few seconds. The melt was stirred effectively by the reaction and the induction field. The melt was poured into copper molds 1 minute after the start of the reaction. A thin skin of slag formed on the melt, but this was caught by the crucible walls during pouring. Typical castings are shown in figures 3(a) to (d).

Hot-Rolling

Specimens listed in table IV were hot-rolled in a 4-high rolling mill with $2\frac{1}{2}$ -inch-diameter working rolls. The ingots were heated in a conventional Globar furnace and reheated after each pass. The first soaking period was 1/2 hour. Soaking time between passes varied from 1/2 to 1/4 hour, depending on the thickness of the specimen. Approximately 3 seconds were required for the removal of the specimen from the furnace to the rolls. The soaking temperature for hot-rolling was

2400° F. After reduction, the specimens were cooled by placing them on a metal sheet in a stream of air. Typical shapes rolled are shown in figures 3(e) and (f).

A typical rolling schedule for the 17.5-percent-aluminum alloy is as follows:

Pass	Reduction per pass, percent
1	2.7
2	6.0
3	8.6
4	12.0
5	15.9

The angle of nip limited the maximum reduction per pass to approximately 16 percent for specimens with thicknesses greater than 1/2 inch.

Heat-Treating

Tables V, VI, and VII list test specimens which were heat-treated. These specimens were heat-treated in air in the as-cast condition. A set of hot-rolled specimens for metallographic examination was also heat-treated (after rolling) for 48 hours at temperatures of 1500°, 1700°, 1800°, 2000°, 2200°, 2300°, 2400°, and 2500° F. These specimens were used to determine whether hot-rolling altered the changes in structure produced by the heat-treatments alone. It was of special interest to determine which phases were present at 2400° F, since this is the temperature at which the 17.5-percent-aluminum alloy had been rolled most successfully.

Machining

Despite the comparative softness of the materials; which was Rockwell A-60 to A-70 (Rockwell C-20 to C-39), it was difficult to machine these alloys because of excessive tool wear. The specimens were therefore ground to shape using silicon carbide wheels.

Specimen Evaluation

Short-time tensile evaluation. - Because of the ease of preparation and suspected low ductility of the alloys, conical-end specimens (fig. 3(g)) were used. These specimens had a 1/4-inch-diameter gage section

and a $1\frac{1}{4}$ -inch gage length. A conventional tensile machine was used, and stress-strain curves were obtained by means of a recording extensometer. For high-temperature evaluations, the specimens and holders were enclosed in a platinum-wound tube furnace and the ductility was measured after fracture. Tables III, V, and VI list specimens which were evaluated by tensile tests.

3121 Creep-rupture evaluation. - Except for the use of conical-end specimens, the method used in creep-rupture evaluation was the same as that employed for the creep-rupture testing of alloys (ref. 5). The extensometer consists of a platinum rod which slides in a platinum tube. The rod is cemented to one end of the specimen gage length, and the tube to the other. Creep is measured by determining the change in distance between scribed marks on the platinum rod and tube, using a cathetometer sighted through a window in the furnace. The creep-rupture tests were run on a 17.5-percent-aluminum alloy at 1350° F. Since it was desired to evaluate alloys that were stable at the test temperature, all specimens were heat-treated at 1500° F for 48 hours before testing.

Structure determination. - A Norelco Geiger counter, X-ray spectrometer having a range of 150° 2θ was used to obtain diffraction patterns for five specimens. Patterns were obtained from solids and powders. Four of the specimens, (14-, 17.5-, 25-, and 30-percent-Al compositions) were in the as-cast condition, and the other (17.5-percent-Al composition) was homogenized at 1500° F for 48 hours. The work was done to confirm the presence of Ni₃Al and NiAl phases.

Thermal-shock evaluation. - Two specimens 2 inches in diameter and 1/4 inch thick of the 17.5-percent-aluminum composition which had been hot-rolled 50 percent at 2400° F were tested in thermal shock. The apparatus (described in ref. 6) for the test consisted of a furnace in which the specimen was heated and an adjacent air-duct into which the specimen could quickly be withdrawn. The quenching air flow could be varied. The first air flow employed was at the rate of 265 feet per second and was later increased to 360 feet per second to obtain more rigorous conditions. The specimens were quenched 25 times from a furnace temperature of 1800° F. This cyclic operation was repeated 25 times at temperatures of 2000°, 2200°, and 2400° F or until failure occurred. The velocities and mass flow in the test simulated those of a jet engine.

Density evaluation. - Densities listed in table III were determined by differential weighing of the specimens in air and water.

Oxidation-resistance evaluation. - Specimens of the 17.5-percent-aluminum composition which had been hot-rolled 50 percent at 2400° F were tested for oxidation resistance at 1500°, 1800°, and 2100° F for

times up to 140 hours. For comparative purposes, a weight-gain curve for commercial Nichrome and total weight gains at the end of 100 hours for S-816 and Inconel X were determined. Specimens measuring approximately 3 by 5/8 by 1/4 inches were suspended on a platinum wire from one arm of an analytical balance into a vertical tube furnace as shown in figure 4. Small openings at the bottom and top of the furnace permitted flow of air through the furnace.

Impact evaluation. - Impact tests were run on the 17.5-percent-aluminum alloy in the as-cast condition. The apparatus is shown in figure 5. The notched specimen was held in a vice, and a hammer of known weight was dropped a known height from a magnetic holder so as to strike the specimen near the end. The height of drop was progressively increased until failure occurred. For the first specimen, several trials were required, but the failure height for subsequent specimens was closely estimated and these specimens received only a few nonfailure impacts. The energy to cause failure was calculated. Both notched and unnotched bars were used, the notched bars to assure fixed location and area of failure. Notched specimens of the 17.5-percent-aluminum alloy which had been tested are shown in figure 6.

For high-temperature tests, the specimens can be resistance heated. As shown in figure 5, electrodes may be clamped against the ends of the specimen to permit current flow. A thermocouple at the notch indicates the temperature, and after thermal equilibrium is reached, the electrodes are quickly withdrawn and the hammer dropped.

RESULTS

Quality of Castings

Castings containing up to 30 percent aluminum were sound and free from excessive porosity. In the case of the stoichiometric NiAl (31.5 percent Al) and the 34-percent-aluminum composition, the violence of the exothermic reaction between the nickel and the aluminum increased to the point where, with the techniques employed, it was no longer possible to produce castings which were sufficiently sound to permit physical evaluation.

Physical Evaluation of Cast Alloys

The room-temperature tensile strengths, hardnesses, and densities of castings have been plotted in figure 7. The observed densities (table III) were less than the calculated densities (using the lattice parameters of ref. 7) by a maximum of 2 percent and an average of 1.5 percent.

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Short-time room-temperature tensile strengths are listed in table III, and stress-strain curves are shown in figure 8 for the as-cast compositions. The 14-percent-aluminum (Ni_3Al) composition possessed moderate strength and ductility. The 17.5-percent composition (Ni_3Al and NiAl) was considerably stronger, and retained some room-temperature ductility. Above 17.5 percent aluminum in the NiAl phase region, the strength of the castings decreased and no evidence of ductility was observed. Castings with up to 30 percent aluminum could, however, be dented by a blow of a sharp-edged instrument with evident local deformation and no cracking. Specimens containing 31.5 and 34 percent aluminum shattered under similar blows.

Hot Formability of Alloys

Another set of castings having the same compositions as those employed for the tensile test and, in addition, 15.5- and 21.5-percent-aluminum alloys, were prepared for the hot-rolling tests listed in table IV. The 17.5-percent-aluminum alloy was readily rollable at 2400°F and could be successfully rolled at 2100°F . Microstructures of the 17.5-percent-aluminum alloy after various conditions of rolling are shown in figure 9. As indicated in table IV, the 28-percent-aluminum alloy was not as readily rollable, but a small amount of hot working was possible at 2600°F . No other compositions could be successfully rolled to any appreciable extent. Since the alloy with 28 percent aluminum did not possess good hot-forming properties and was definitely inferior to the 17.5-percent-aluminum alloy in room-temperature properties, the 17.5-percent-aluminum composition was investigated further.

Physical Properties of 17.5-Percent-Aluminum Alloy

Tensile strengths. - Room- and elevated-temperature tensile strengths and elongations for the 17.5-percent-aluminum alloy in the as-cast, homogenized, hot-rolled, and alloyed condition are listed in tables V and VI.

Grain refinement. - Slight amounts of alloying constituents such as 0.5 percent molybdenum, 0.3 percent titanium, and 0.05 percent boron were added to the 17.5-percent-aluminum alloy as grain refiners. The change in the grain size of the alloy produced by these additions is shown in figure 10.

Stress-rupture and creep properties. - Stress-rupture properties are plotted in figure 11. Properties of Inconel (extrapolated data, ref. 8) and Inconel X (ref. 9) are included for comparative purposes.

Creep curves for the 17.5-percent-aluminum alloy are plotted in figure 12. The creep rates (in second-stage creep) at 10,000 and 12,500 pounds per square inch are 0.002 and 0.007 inch per inch per hour, respectively. For higher stresses, the creep rates are excessive.

Hardness. - The hardness results are shown in table VII and are plotted in figure 13. All the specimens on the continuous curve were water quenched. The specimen which was air cooled from 2500° F showed a considerable drop in hardness compared with that of the specimen which had been water quenched from the same temperature.

Impact strength. - At room temperature, the impact strengths of notched specimens were found to be 3.62 inch-pounds, whereas unnotched-specimen strengths were over 10 inch-pounds. Thus, the alloy is definitely notch sensitive. Comparative data for other materials are given in the following table:

Material	Impact strength, in.-lb
Ni-Al(82.5-17.5) ^a	3.52
Ni-Al(82.5-17.5) ^b	3.72
	10.18
K-152B ^a	10.46
	2.1
X-40 ^a	48.05

^aNotched.

^bUnnotched.

Thermal-shock resistance. - Of the two specimens tested in thermal shock, one specimen withstood 100 cycles (25 cycles at 1800°, 2000°, 2200°, and 2400° F) at the low air flow (265 ft/sec) and another 100 cycles at the higher air flow (360 ft/sec) at the same temperature. A second specimen was quenched at 2400° F in the high air flow and failed after 48 cycles. Small (1/2- by 1/2- by 1/2-in.) specimens used in the homogenization investigation did not fail when quenched in water from temperatures of 2200° F and below.

Oxidation properties. - Results of the oxidation evaluation have been plotted in figure 14. A weight gain of 1.28 milligrams per square centimeter occurred at 2100° F after 140 hours. Maximum weight gains of 0.440 and 0.228 milligram per square centimeter occurred at temperatures of 1800° and 1500° F, respectively, after periods of approximately 100 hours. The oxide layer did not flake off and was very adherent. A weight-gain curve for Nichrome (a nickel-base alloy containing 20 percent

chromium) at 1800° F is shown in figure 14. Also shown are 100-hour total weight-gain points at 1500° F for the high-temperature alloys S-816 and Inconel X.

Microstructure of 17.5-Percent-Aluminum Alloy

Phase identification. - The phases Ni_3Al and NiAl were identified by X-ray techniques. Lattice parameters obtained are as follows:

NiAl - 2.860 kxunits

Ni_3Al - 3.573 kxunits

The lattice parameters from compositions within the NiAl phase region were found to agree with A.S.T.M. card data for NiAl if corrected as indicated in reference 7 for the variation in aluminum content throughout the phase region. The lattice parameters determined for the Ni_3Al region were in agreement with those of reference 7. The 17.5-percent-aluminum composition after heat-treating 48 hours at 1500° F showed both phases to be present. Both phases, however, could not be identified in the as-cast condition.

Results of solution-treatment. - Microstructures of specimens heat-treated at 1500°, 1700°, 1800°, 2000°, 2200°, 2300°, 2400°, and 2500° F are shown in figures 15 and 16. Descriptions of the various microstructures are given in table VII. The as-cast structure was a fine lamellar structure. Increasing the solution-treatment temperature from 1500° to 1800° F progressively coarsened the structure. At 2000° F, the white phase (Ni_3Al) appeared to have decreased. At 2200° F, subgrain formation takes place. Almost all the Ni_3Al at the grain boundaries dissolved into the matrix at 2300° F, and a plate-like structure was evident. At 2400° and 2500° F, the Ni_3Al phase is indicated by the equilibrium diagram to be completely dissolved into the NiAl phase. The photomicrographs show the matrix to be acicular (figs. 15(g) to (i), and figs. 16(i) and (j)) and the matrix might be termed a martensite-like NiAl phase (partially transformed NiAl phase). In grain boundary areas, Ni_3Al appears to have formed (figs. 16(g) to (i)), and in figures 15(g) to (j), Ni_3Al appears to have formed in a Widmanstätten pattern. Upon air cooling from 2500° F, the precipitation of Ni_3Al occurs in a Widmanstätten form.

Microstructures of specimens which had been hot-rolled 50 percent at 2400° F and heat-treated at temperatures of 1500°, 1700°, 1800°, 2000°, 2200°, 2300°, 2400°, and 2500° F are not shown. The structures resulting from the thermal treatments were the same for both the as-cast

and hot-rolled conditions; therefore, since all the hot-rolled material was large-grained, the changes observed in the grain size of the as-cast structure would be more apparent.

DISCUSSION OF RESULTS

Casting Method

It has been very difficult to cast alloys in the NiAl range of the nickel-aluminum system. Reference 2 reports that cast bars of NiAl literally fell apart in handling, apparently from lack of cohesion between the grains. Castings of the 30-percent-aluminum composition made for this investigation had tensile strengths as high as 16,300 pounds per square inch at room temperature. This composition is very close to the stoichiometric NiAl composition (fig. 1).

An important difference between the casting method used in this study and those used in previous investigations (ref. 2) is that fluxing agents are not used in the method developed here. Since the reaction is violently exothermic and very high temperatures may be attained, reactions may occur between the fluxing compounds and the molten metal. If the product of such reactions is retained in the grain boundaries, it may be the cause of the lack of cohesion between the grains and the extreme brittleness.

The casting method was satisfactory for the production of sound castings weighing up to $1\frac{1}{2}$ pounds. Difficulties may be encountered in the production of larger castings because of the extremely exothermic nature of the reaction between nickel and aluminum. The method used in this investigation in which solid nickel reacts with molten aluminum should tend to keep the final temperature considerably lower than when aluminum is added to molten nickel, a technique sometimes employed. Because the violence of the reaction increases with increasing aluminum content, it is difficult to make porosity-free castings with aluminum contents over 30 percent. However, casting into a ceramic mold, which would keep the material molten until the reaction subsides, may produce sound castings. The grain size in such a case would be very coarse.

Actual compositions agree closely with intended compositions in table II. Evidently, little loss of aluminum occurred during melting, which may be attributed to the short time the heats were held molten.

Properties of Ni₃Al

Literature giving properties of as-cast Ni₃Al was unavailable. In reference 4, it is stated that Ni₃Al is a hard, brittle material. The

present investigation has proved Ni_3Al to have some ductility. The as-cast specimens exhibited room-temperature tensile strengths of approximately 48,000 pounds per square inch with 2.5- to 6.3-percent elongation. However, at 1500°F , the strength and ductility of this intermetallic compound decreased to 37,500 pounds per square inch and 1.3-percent elongation. Two attempts were made to roll the alloy at 2000°F and one at 2400°F , and in all cases the material was hot short.

Properties of NiAl

An alloy of the NiAl composition (25 percent Al) had a room-temperature strength of approximately 22,000 pounds per square inch with no measurable ductility. At 1500°F , the strength of this alloy had increased to 31,800 pounds per square inch with a 3.8-percent elongation (table VI). This increased strength may be the result of the increased plasticity of the material at 1500°F and the lessening effect of bending during the tensile test.

Properties of 17.5-Percent-Aluminum Alloy

The 17.5-percent-aluminum alloy had the most outstanding combination of hot formability, strength, room-temperature and 1500°F ductility, oxidation resistance, and thermal-shock behavior of the alloys studied.

Tensile strength. - The strength of the as-cast 17.5-percent-aluminum alloy at room temperature was approximately 80,000 pounds per square inch (table V). In one of the two test specimens, a definite measured elongation of 1.2 percent was determined; the other specimen had no measurable elongation after fracture. However, the latter specimen did show both elastic and plastic elongation during testing as measured with an extensometer. Because of the reported brittleness of the NiAl phase, even limited ductility in this alloy, which has an NiAl phase matrix, is significant. At 1500°F , the tensile strength of this alloy was approximately 50,000 pounds per square inch with definite evidence of plasticity; the elongation and reduction in area were 2.9 and 3.3 percent, respectively.

Effect of mechanical working on strength. - Hot-rolling 25 and 50 percent at 2400°F produced 1500°F tensile strengths of 44,800 and 50,850 pounds per square inch with elongations of 1.9 and 3.9 percent. A single specimen, which was hot-rolled 50 percent (included in table VI for comparison purposes) and tested at 1800°F , had a tensile strength of 21,000 pounds per square inch and an elongation of 25 percent.

Hot-rolling is a useful shaping technique but apparently adds little to the properties of the alloy. There was a slight strength loss at

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25-percent reduction by rolling, but at 50-percent reduction, the strength increased to approximately that of the as-cast material. Ductility was appreciably increased (3.5 to 4.4 percent) compared with the as-cast material in cases in which specimens were reduced 50 percent (tables V and VI). From figures 9(a) and (b), it is evident that the hot-rolled material was much more homogeneous than, and did not have the segregations of, the as-cast material. The size and orientation of the white phase (Ni_3Al) also changed. The dispersion, size, and orientation of the white phase may have a marked effect on the ductility of the 17.5-percent-aluminum alloy.

Effects of grain-refining agents. - The addition of grain-refining agents to the 17.5-percent-aluminum alloy did not improve strength properties of the basic alloy. Titanium, molybdenum, and boron had a marked effect on grain size (fig. 10). Figure 10(b) shows that titanium refined the coarse columnar as-cast structure of figure 10(a). The addition of 0.05 percent boron (fig. 10(c)), had some effect on the grain size but not as great as that of titanium, since the grain structure was not equiaxed but of a finer columnar nature. A 0.5-percent-molybdenum addition (fig. 10(d)) produced an equiaxed structure comparable to that of titanium. Only boron produced a strengthening effect on the as-cast material at room temperature (table V). However, at 1500° F (table VI), the boron-containing material had the lowest strength, while 0.3 percent titanium increased the strength from 52,900 to 56,000 pounds per square inch. No appreciable effect of grain size on short-time strength was indicated from these tests.

Stress-rupture. - The stress-rupture properties plotted in figure 11 show that the 17.5-percent-aluminum alloy is slightly better than Inconel but is inferior to Inconel X. It should be noted that Inconel X is a precipitation-hardening alloy, while nothing was added to the 17.5-percent-aluminum alloy to improve the strength. Inconel has a short-time strength of 27,000 pounds per square inch at 1400° F and a strength of approximately 20,000 pounds per square inch at 1500° F (extrapolated data, ref. 8). The 17.5-percent-aluminum alloy has a strength of about 50,000 pounds per square inch at 1500° F. Although the strength of the 17.5-percent-aluminum alloy is $2\frac{1}{2}$ times greater than that of Inconel at 1500° F, the alloy is just slightly better in creep-rupture at 1350° F. In the case of Inconel, 14.5 percent chromium is added and remains in solid solution. In the 17.5-percent-aluminum alloy, the aluminum content is beyond that of the solid-solution range, and a two-phase mixture appears (Ni_3Al and NiAl). An absence of a finely dispersed precipitate may be the reason for the high creep rates of the alloy (fig. 12). If the creep rates can be reduced by slight alloying additions which form a precipitation-hardening phase, a useful alloy may result for creep-rupture applications.

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Impact strength. - It was previously shown that the 17.5-percent-aluminum alloy is very much weaker in room-temperature impact strength than alloy X-40 and only slightly stronger than the cermet K-152B. However, there is evidence that the 17.5-percent-aluminum alloy does not behave as a cermet in impact even though the measured impact values would so indicate. Figure 6 shows that upon impact, the specimens have not fractured completely, whereas a cermet specimen given the same blow would have been severed. This behavior is analogous to alloys subjected to the impact test. Therefore, the alloy has some inherent ductility.

Thermal shock. - The ability of a material to withstand 100 cycles at the low air flow in the thermal-shock rig described is believed to be adequate for a turbine-blade material. It was shown that the 17.5-percent-aluminum alloy resisted over 200 cycles of thermal shock testing in an apparatus designed to simulate jet-engine blow-out conditions. If this alloy were used as a base material for a turbine-blade alloy, it could be expected to resist thermal shock satisfactorily.

Oxidation resistance. - The weight-gain curves of figure 14 show the 17.5-percent-aluminum alloy to be slightly better in oxidation resistance than Nichrome at 1800° F. Nichrome is widely used as a heating element because of its oxidation resistance, as well as its electrical properties. On the basis of oxidation resistance, the 17.5-percent-aluminum alloy might also be useful in this capacity. The substitution of cheaper and more available aluminum for chromium may make it commercially feasible. It is also shown that the 17.5-percent-aluminum alloy is superior in oxidation resistance to the high-temperature alloys S-816 and Inconel X. Total weight gains after 100 hours at 1500° F of 0.603 and 0.522 milligram per square centimeter for S-816 and Inconel X, respectively, are higher than the maximum 100-hour weight gains at 1800° F for the 17.5-percent-aluminum alloy.

Microstructure of 17.5-Percent-Aluminum Alloy

Similarity to brass. - Most of the studies on microstructures were concentrated on the 17.5-percent-aluminum alloy. Since this alloy had the most promising properties, as much information as possible was obtained concerning the two-phase structure. The 17.5-percent-aluminum composition is in a phase field similar to that of the alpha-beta brass field of the copper-zinc equilibrium diagram. The behavior of these copper-zinc alloys is well known (ref. 10), and microstructures of copper-zinc alloys have striking similarities to structures of some of the 17.5-percent-aluminum specimens.

As-cast structure. - The as-cast structure was determined metallographically and consisted of two phases, Ni_3Al and NiAl (fig. 1). Both phases were identified using specimens which has been homogenized at

1500° F since X-ray diffraction proved impractical on as-cast specimens. The phase diagram of the nickel-aluminum system (réf. 11) has been substantiated by an extensive X-ray study (ref. 7); consequently, metallographic investigations alone were relied upon in the analysis of the structures.

Effect of solution-treating temperature. - The heat-treating temperatures were raised progressively to determine how the as-cast microstructures were altered by heat-treatment and to determine at what temperature solution of the Ni₃Al phase into the NiAl phase occurred. As the temperature of heat-treatment increases, elimination of as-cast chemical heterogeneities, changes in composition of the Ni₃Al and NiAl phases, and changes in the relative quantities of each phase would be expected. The change in the relative quantities of each phase is evident in the photomicrographs of figure 15. Changes in composition (such as coring) were not observed metallographically.

From 1500° to 1800° F, the hardness decreased with further agglomeration of the Ni₃Al phase and increased as the plate-like structure became evident (figs. 13 and 16). The martensite-like NiAl phase was the hardest structure. Precipitation of the Ni₃Al phase in a Widmanstätten structure caused a drop in hardness, as is evident in the air-cooled structure (table VII).

At 2200° F, some of the original Ni₃Al phase remained in the grain boundary and scattered randomly in the structure. The matrices of the grains, however, apparently transformed to the NiAl phase at this temperature. During the transformation of the matrix, recrystallization occurred, and grains were nucleated within the original boundary areas. These grains (subgrains) apparently transformed to a martensite-like structure upon water quenching, and resembled the martensite of reference 12.

At 2300° F, the solution of the Ni₃Al phase at the grain boundary was complete, permitting recrystallization and grain growth of the entire NiAl phase. During water quenching, a more acicular martensite-like structure formed (figs. 15(g) and 16(g)); the Ni₃Al precipitated at the grain boundaries differed from the martensite-like structure.

Heat-treating at 2400° and 2500° F caused complete solution of the Ni₃Al phase into the NiAl matrix, but cooling conditions were such that a Widmanstätten phase was nucleated at the grain boundaries with a metastable (martensite-like) phase within the grains (figs. 15(h) and (i)).

Effect of cooling rate. - Intermediate quenching treatment (air blast) resulted in a fine Widmanstätten precipitated within the grains and at the grain boundaries, and also a martensite-like structure within

the grains (fig. 15(j)). Upon air cooling, a fine lamellar structure (coarse Widmanstätten) was obtained (figs. 15(k) and 16(k)). Behavior of specimens heated at 2400° and 2500° F was similar to that of 2300° F except that a fine Widmanstätten precipitate formed at the grain boundaries.

Effect of hot-rolling. - In the phase diagram of figure 1, it may be seen that at the rolling temperature, 2400° F, all phases of the 17.5-percent-aluminum alloy have transformed to NiAl. The precipitation of Ni₃Al was very rapid upon cooling from temperatures between 2400° and 2500° F. The Ni₃Al was observed to partially transform from NiAl upon water quenching. About 3 seconds were required to remove the ingots to be rolled from the furnace to the rolls. Even though the cold rolls acted as quenching medium, the centers of the ingots were probably untransformed NiAl. This was confirmed by microscopic examination of polished specimens of rolled material, which disclosed large islands of martensite-like NiAl, as shown in figure 9(c). The martensite-like acicular structure is more evident in figure 9(d), which is comparable to the homogenized as-cast structure of figure 16(i). The fact that the alloy was able to sustain a 16-percent reduction indicated that the NiAl phase which was untransformed is plastic.

GENERAL REMARKS

It was mentioned that the martensite-like transformation in the 17.5-percent-aluminum alloy appears to be very similar to the transformation which occurs in alpha-beta brasses. In reference 10, it is stated that a transformation of this nature in the brass system had no commercial application. However, in the nickel-aluminum system, it is possible that an alloy of such a basic composition as the 17.5-percent-aluminum alloy could be used as a basic composition for a high-temperature alloy. If alloying additions were made to this basic composition in such a manner that the martensite-like transformation could occur and furnish nucleation sites for precipitation of inter-metallic compounds or carbides, a possible high-strength high-temperature alloy could be evolved.

Reference 13 states that optimum high-temperature properties were obtained in a wrought cobalt-base alloy by a double aging which produced randomly distributed precipitates in the microstructure. The possibility that a martensitic transformation might facilitate evenly distributed precipitation of carbides or borides in an alloy of the basic 17.5-percent-aluminum composition suggests that this alloy is worthy of consideration as a basic composition for other high-temperature alloys.

SUMMARY OF RESULTS

Several properties of nickel-aluminum alloys ranging in aluminum content from 14 to 34 percent were determined. The following results were obtained:

1. Alloys containing up to 30 percent aluminum were satisfactorily cast despite the violently exothermic reaction between the constituents. The melting practice was most important and consisted of reacting molten aluminum with solid nickel, which tended to keep the final temperature of the melt down.

2. The Ni_3Al intermetallic compound, represented by a cast alloy containing 14 percent aluminum, had strengths of 48,450 pounds per square inch in room-temperature tensile tests, and 37,500 pounds per square inch at 1500°F . Ductility (total elongation) ranged from 2.5 to 6.3 percent at room temperature, and was 1.3 percent at 1500°F . Thus, the Ni_3Al phase possesses moderate strength and some ductility.

3. The NiAl cast intermetallic compounds investigated ranged in composition from 25 to 30 percent aluminum. Room-temperature tensile strengths were highest in the 25-percent-aluminum alloy, averaging 22,000 pounds per square inch, and lowest in the 30-percent-aluminum alloy, averaging 14,000 pounds per square inch. The ductility (total elongation) of the 25-percent-aluminum alloy was zero at room temperature and 3.8 percent at 1500°F .

The NiAl phase had lower strength and ductility than the Ni_3Al phase. However, the ductility at 1500°F indicated that this phase is not as brittle as is indicated in the literature.

4. The strengths of the as-cast 17.5-percent-aluminum alloy which contained the two phases Ni_3Al and NiAl were the highest of the compositions cast. The room-temperature tensile strength was 79,600 pounds per square inch in a specimen which elongated 1.2 percent. A homogenization treatment at 2400°F of an as-cast alloy increased the strength to 94,250 pounds per square inch with no elongation. The grain refiners titanium and molybdenum did not appreciably increase the room-temperature tensile strength of the as-cast alloy, but boron in a small quantity (0.05 percent) increased the as-cast strength to 92,600 pounds per square inch.

5. Of the cast alloys studied, only the 17.5- and 28-percent-aluminum alloys could be hot-rolled, the former at 2400°F and the latter at 2600°F . The room-temperature tensile strengths of the 17.5-percent-aluminum alloy rolled 10 percent was 73,400 pounds per square inch, whereas with 50-percent rolling the strength was 86,300 pounds per square inch. After breaking up the as-cast segregations, it was possible to roll the 17.5-percent-aluminum alloy as much as 15.9 percent in a single pass at 2400°F .

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6. The 17.5-percent-aluminum alloy was the only alloy tested in creep-rupture at 1350° F. The 100-hour strength of the alloy was 14,000 pounds per square inch, while the 1000-hour strength was 8500 pounds per square inch. Creep rates for the alloy were high. The creep-rupture strength of the alloy at this temperature is comparable to Inconel, a nonprecipitation-hardening solid-solution alloy. The 17.5-percent-aluminum alloy possesses moderate impact strength, good thermal shock behavior, and outstanding oxidation resistance.

7. Cast 17.5-percent-aluminum alloy specimens were heat-treated in the temperature range 1500° to 2500° F. The following changes in microstructure were obtained:

The as-cast alloy consisted of a coarse Widmanstätten structure of two phases, Ni₃Al and NiAl. As the temperature of heat-treatment increased from 1500° to 2000° F, changes in the relative quantities of each phase occurred, with agglomeration of the Ni₃Al phase. At 2200° F, the matrices of the grains transformed to NiAl with simultaneous recrystallization. During water quenching, these grains (subgrains) transformed to a martensite-like structure. Recrystallization and grain growth took place at 2300° F with the structure transforming to an acicular martensite-like structure during water quenching. Heat-treating at 2400° and 2500° F followed by water quenching resulted in a Widmanstätten structure nucleated at the grain boundaries with a metastable (martensite-like) phase within the grains. Upon air cooling, a fine lamellar structure (coarse Widmanstätten) was obtained.

CONCLUDING REMARKS

The martensite-like transformation of the high-temperature NiAl phase of the 17.5-percent-aluminum alloy was very rapid, even during water quenching. This rapid transformation may be useful in high-temperature applications. Additions made to an alloy having the 17.5-percent-aluminum alloy as a base, and using the martensite-like transformation to furnish nucleation sites for the precipitation of intermetallic compounds or carbides, may result in a high-creep-strength high-temperature alloy. It is possible that random evenly-distributed precipitates in the microstructure, which are desirable for high-temperature strength, may be obtained by aging a precipitate in these nucleation sites. The 17.5-percent-aluminum alloys may be worthy of consideration as a basic composition in the search for replacement of critical cobalt-base alloys by nickel-base alloys.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, June 17, 1954

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TABLE I. - CHEMICAL ANALYSIS OF RAW MATERIALS

Material	Composition, percent				
	Al	Fe	Ni	Mn	Silica
Electrolytic Ni	-----	-----	99.95	-----	-----
2S Al	99.0	0.58	-----	0.03	^a 0.4
High purity Al	99.85	-----	-----	-----	-----

^aOr less determined by difference.

TABLE II. - CHEMICAL ANALYSIS OF SPECIMENS

Intended composition, percent	Actual composition, percent				
	Al	Fe	Ni	C	Total
86 Ni + 14 Al	14.16	0.10	85.76	-----	100.02
82.5 Ni + 17.5 Al	17.61	.15	82.15	0.02	99.93
75 Ni + 25 Al	25.21	.19	74.71	-----	100.11
72 Ni + 28 Al	28.50	.24	71.31	-----	100.05
70 Ni + 30 Al	29.93	.28	69.49	-----	99.70

TABLE III. - TENSILE PROPERTIES OF CAST NICKEL-ALUMINUM

ALLOYS AT ROOM TEMPERATURE

Chemical composition, percent		Phases present	Tensile strength, psi	Elongation, percent	Hardness, Rockwell A	Density, g/ml
Ni	Al					
86	14	Ni ₃ Al	50,600 45,900	6.3 2.5	62	7.39
82.5	17.5	Ni ₃ Al, NiAl	83,800 79,600	0 1.2	69	6.99
75	25	NiAl (a) (a)	24,100 20,200 22,100 19,300	0 0 0 0	72	6.33
72	28	NiAl	20,000 22,400	0 0	71	6.12
70	30	NiAl	16,300 12,500	0 0		5.98
68.5	31.5	NiAl	Unsound ingot	Unsound ingot	Unsound ingot	5.89
66	34	NiAl	Unsound ingot	Unsound ingot	Unsound ingot	

^a 0.3 Percent Ti added as grain refiner.

TABLE IV. - HOT-ROLLING BEHAVIOR OF NICKEL-ALUMINUM ALLOYS

Specimen	Chemical composition, percent		Phases at room temperature	Rolling temperature, °F	Rolling behavior	Passes before cracking	Reduction during pass, percent	Remarks
	Ni	Al						
1	86	14	Ni ₃ Al	2000	Poor	1	3.7	Large cracks
2				2000	Poor	2	.8	Large cracks
3				2400	Poor	1	1.4	Very bad cracks at sides
4	84.5	15.5	Ni ₃ Al	2400	Poor	1	3.1	Cracks on one side
5	82.5	17.5	Ni ₃ Al, NiAl	2400	Good		15.9 (Maximum reduction occurred in 5th pass)	Maximum reduction limited by angle of nip, did not crack
6				2200	Rollable		3.9 (Maximum reduction occurred in 6th pass)	Did not crack
7				2100	Rollable		2.1 (Maximum reduction occurred in 1st pass)	Did not crack
8	78.5	21.5	Ni ₃ Al, NiAl	2400	Poor	1	3.1	Small cracks both sides
9	75	25	NiAl	2400	Poor	1	2.8	Small cracks both sides
10				2600	Poor	2	1.3	Bad side cracks
11	72	28	NiAl	2600	Rollable		8.3 (Maximum reduction occurred in 9th pass)	Did not crack
12	70	30	NiAl	2600	Doubtful	3	1.9	Cracks

TABLE V. - ROOM-TEMPERATURE TENSILE PROPERTIES OF 17.5-PERCENT-

ALUMINUM ALLOY
[Original condition, as-cast.]

Heat-treatment	Addition	Tensile strength, psi	Extensometer elongation, percent	Measured elongation, percent
None	None	83,800	0.6	0
		79,600	.6	1.2
Homogenized 16 hr, 2400° F	None	94,250	0	---
Homogenized 48 hr, 1500° F	None	55,800	0	---
Homogenized 48 hr, 2000° F	None	33,400	0	0
Rolled 10 percent, 2400° F	None	73,400	0	---
Rolled 25 percent, 2400° F	None	73,050	0.8	---
		80,500	.6	---
Rolled 50 percent, 2400° F	None	86,300	0	---
		82,700	---	---
Rolled 25 percent, 2400° F, 13 percent, 2200° F	None	^a 69,600	0	0
None	0.5 percent Mo	83,800	0.6	1.5
None	5.0 percent Mo	88,600	0.2	---
Rolled 34 percent, 2400° F	5.0 percent Mo	84,400	0.4	---
None	0.3 percent Ti	84,300	0.4	---
None	0.05 percent B	92,600	---	1.5

^aPure aluminum.

TABLE VI. - TENSILE PROPERTIES OF 17.5-PERCENT-

ALUMINUM ALLOY AT 1500° F

[Original condition, as-cast]

Heat-treatment	Addition	Tensile strength, psi	Total elongation, percent	Area reduction, percent
None	None	52,900	2.9	3.3
Homogenized 16 hr, 2400° F	None	37,600	0.9	0.9
Homogenized 48 hr, 1500° F	None	44,150	1.3	1.0
Homogenized 48 hr, 2000° F	None	55,250	2.5	1.4
Rolled 25 percent, 2400° F	None	48,600	2.0	1.9
		41,000	1.8	1.2
Rolled 50 percent, 2400° F	None	50,000	4.4	5.2
		51,700	3.5	3.6
		^a 21,000	25.0	19.8
Rolled 25 percent, 2400° F, 13 percent, 2200° F	None	^b 40,400	1.3	
None	0.5 percent Mo	52,600	3.7	3.8
None	5.0 percent Mo	51,200	2.0	2.8
Rolled 34 percent, 2400° F	5.0 percent Mo	55,000	2.8	3.2
None	0.3 percent Ti	56,000	3.9	4.1
None	0.05 percent B	43,800	2.5	2.8
(c)	None	37,500	1.3	0
(d)	0.3 percent Ti	31,800	3.8	4.6

^aTest temperature, 1800° F.^bPure aluminum.^c14-Percent-aluminum alloy.^d25-Percent-aluminum alloy.

TABLE VII. - EFFECT OF 48-HOUR HOMOGENIZATION TREATMENTS ON MICROSTRUCTURE
OF 17.5-PERCENT-ALUMINUM ALLOY

Solution-treating temperature, OF	Hardness, Rockwell A	Figure	Appearance of phases	Structure	Effect on grain size
As-cast	69.0	15(a) 16(a)	White Ni_3Al phase, gray NiAl matrix	Coarse Widmanstätten	
1500	71.5	15(b) 16(b)	White agglomerated Ni_3Al phase, dark NiAl matrix	Coarse lamellar	None
1700	66.3	15(c) 16(c)	Formation of Ni_3Al platelets in NiAl matrix	Coarse lamellar	None
1800	66.0	15(d) 16(d)	Apparent solution of Ni_3Al plates into NiAl matrix	Very coarse agglomerated Ni_3Al	None
2000	67.6	15(e) 16(e)	Solution of Ni_3Al platelets, agglomerated Ni_3Al	Coarse Widmanstätten structure	None
2200	75.4	15(f) 16(f)	Plate formation in NiAl matrix, heavy agglomeration of Ni_3Al phase in grain boundaries	Orientation of plates in grains, outline subgrain formation	Subgrain formation, beginning of recrystallization
2300	81.1	15(g) 16(g)	Disappearance of most of agglomerated Ni_3Al phase from grain boundaries, complete plate formation within grains	Plate-like structure	Large grain recrystallized
2400	82.4	15(h) 16(h)	Solution of Ni_3Al phase into NiAl matrix to form martensite-like NiAl matrix, nucleation of Ni_3Al phase at grain boundaries.	Acicular NiAl	Large grains
2500	82.2	15(i) 16(i)	Martensite-like NiAl matrix, Ni_3Al in grain boundaries	Acicular NiAl , very little Ni_3Al precipitation	Large grains
2500 Air blast	80.1	15(j) 16(j)	Martensite-like NiAl matrix, Ni_3Al in Widmanstätten patterns	Acicular NiAl and Ni_3Al Widmanstätten structure	Large grains
2500 Air cool	70.4	15(k) 16(k)	Complete transformation of NiAl phase to two-phase mixture (Ni_3Al , NiAl)	Coarse Widmanstätten	Large grains

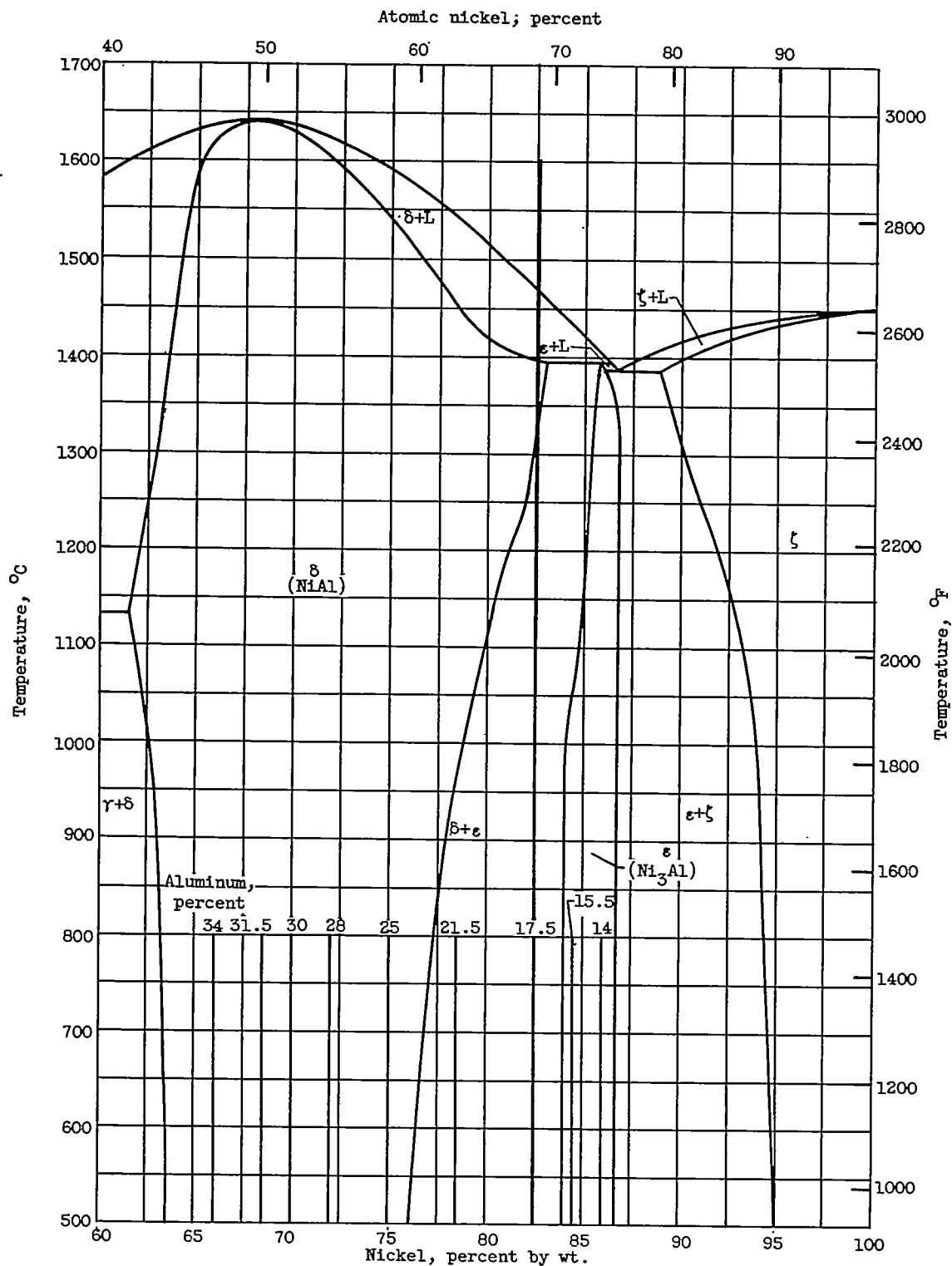
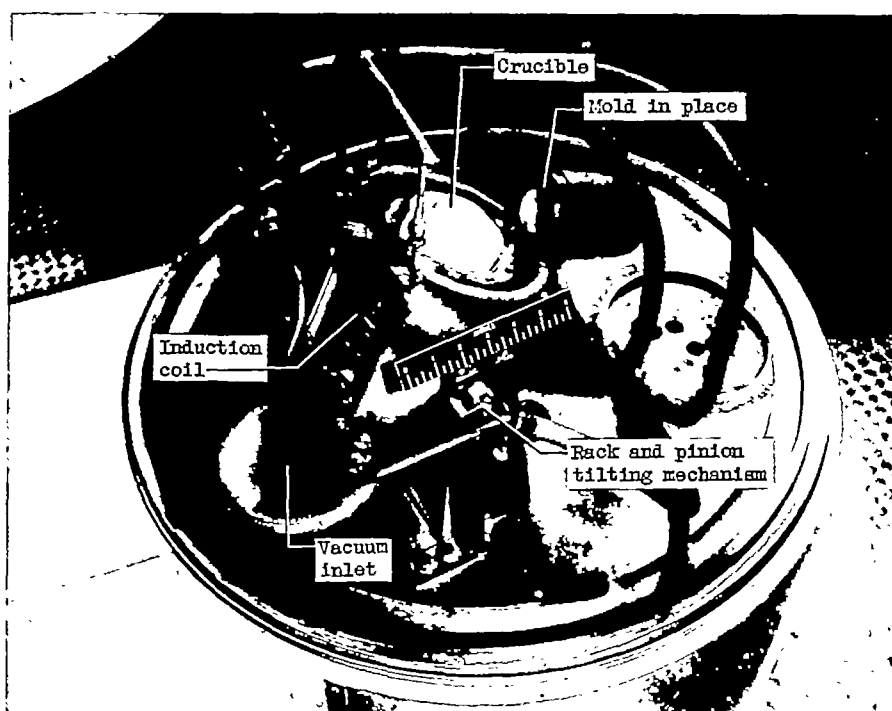
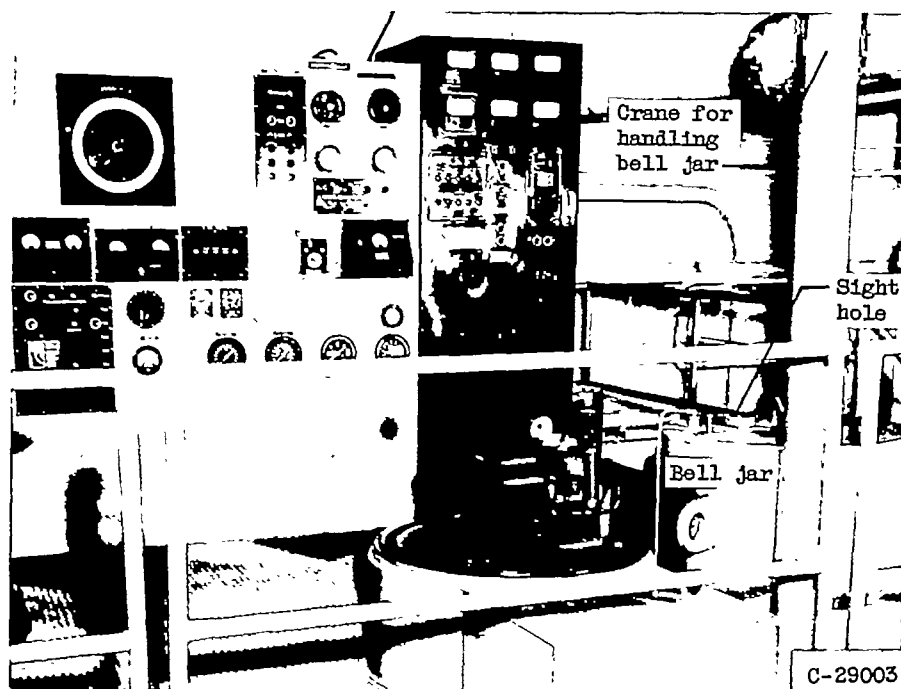


Figure 1. - Enlarged view of area under investigation taken from phase diagram of nickel-aluminum system (ref. 8, p. 1164).



(a) Crucible and mold unit partly tilted toward pouring position.

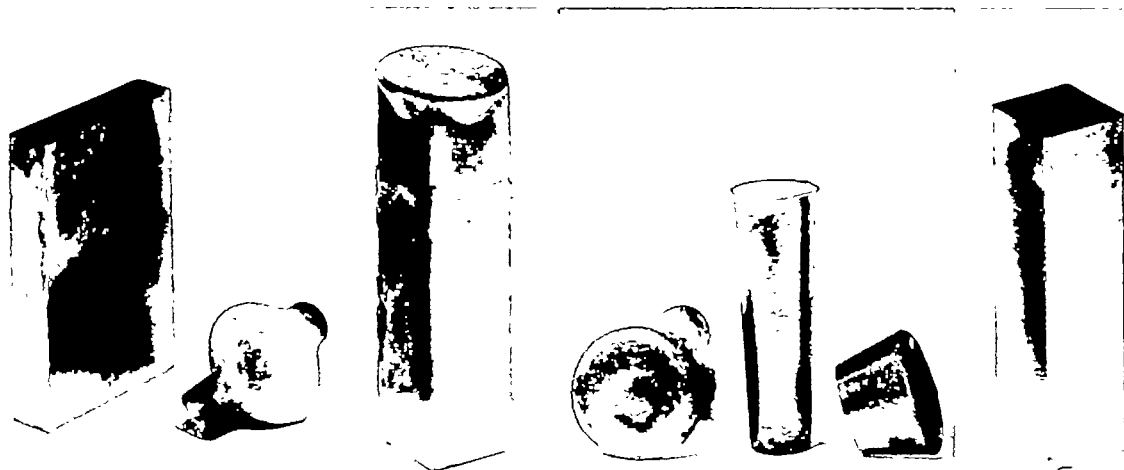


(b) Bell jar removed; control equipment in background.

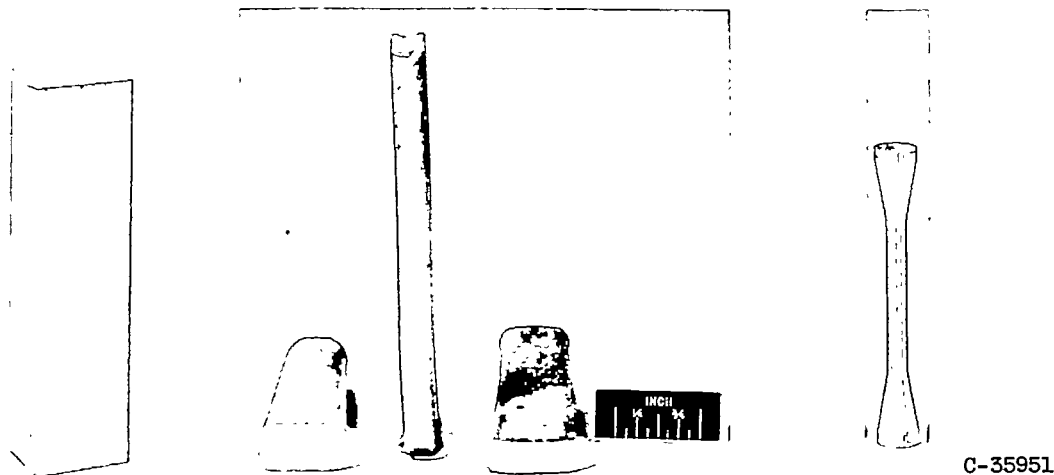
Figure 2. - Vacuum furnace used in casting of nickel-aluminum alloys.

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(a) 1/2- By 2-inch ingot. (b) 1- By 1-inch (c) 1-Inch round tapered ingot. (d) 1- By 1-inch ingot without hot top.



(e) 50-Percent reduction from 1- by 1-inch ingot

(f) 25-and 50-Percent reduction from 1-inch round tapered ingot which had flats ground for rolling.

(g) Ground stress-rupture bar.

Figure 3. - Typical nickel-aluminum specimens.

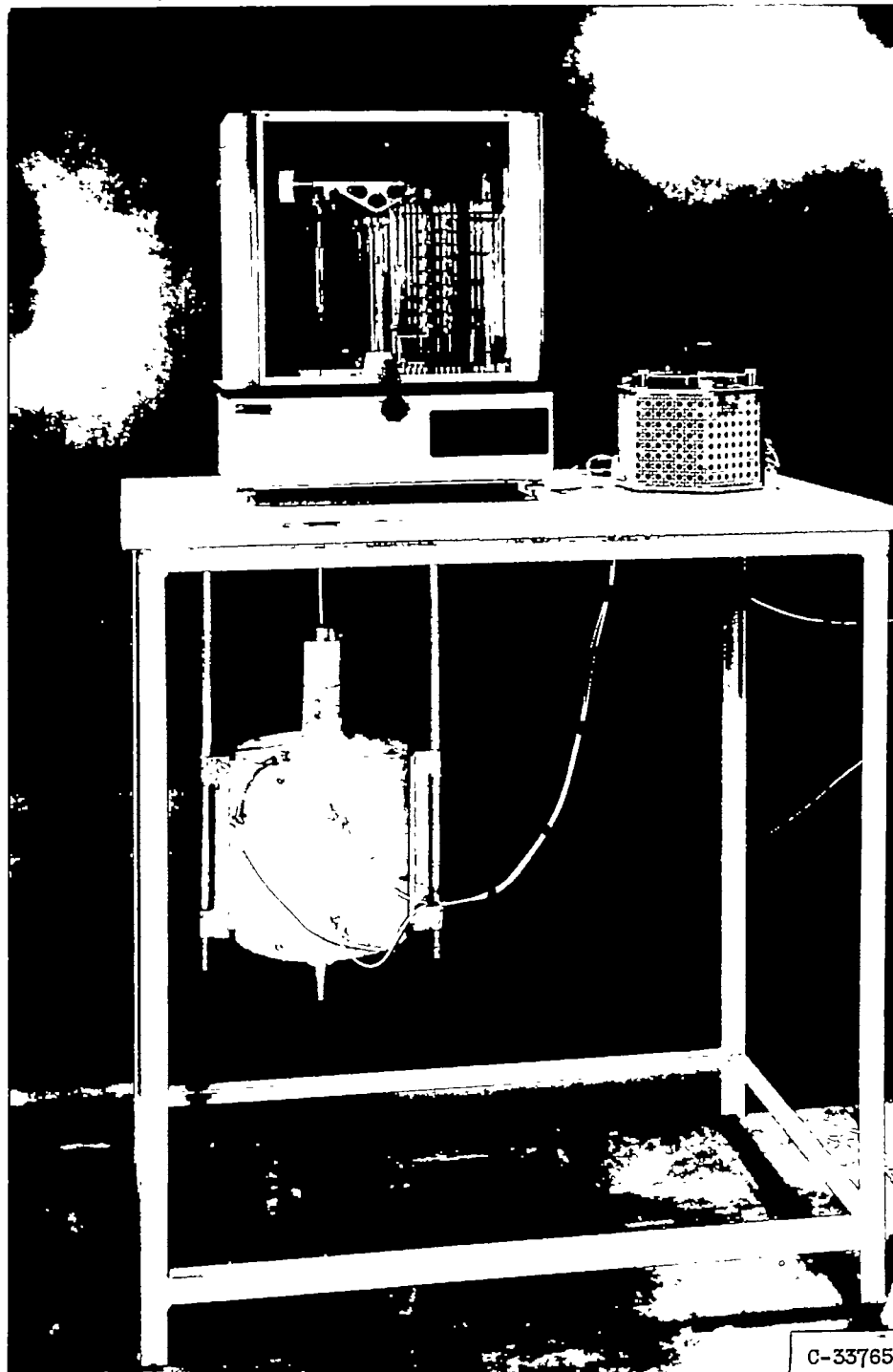


Figure 4. - Furnace and analytical balance for measuring oxidation resistance.

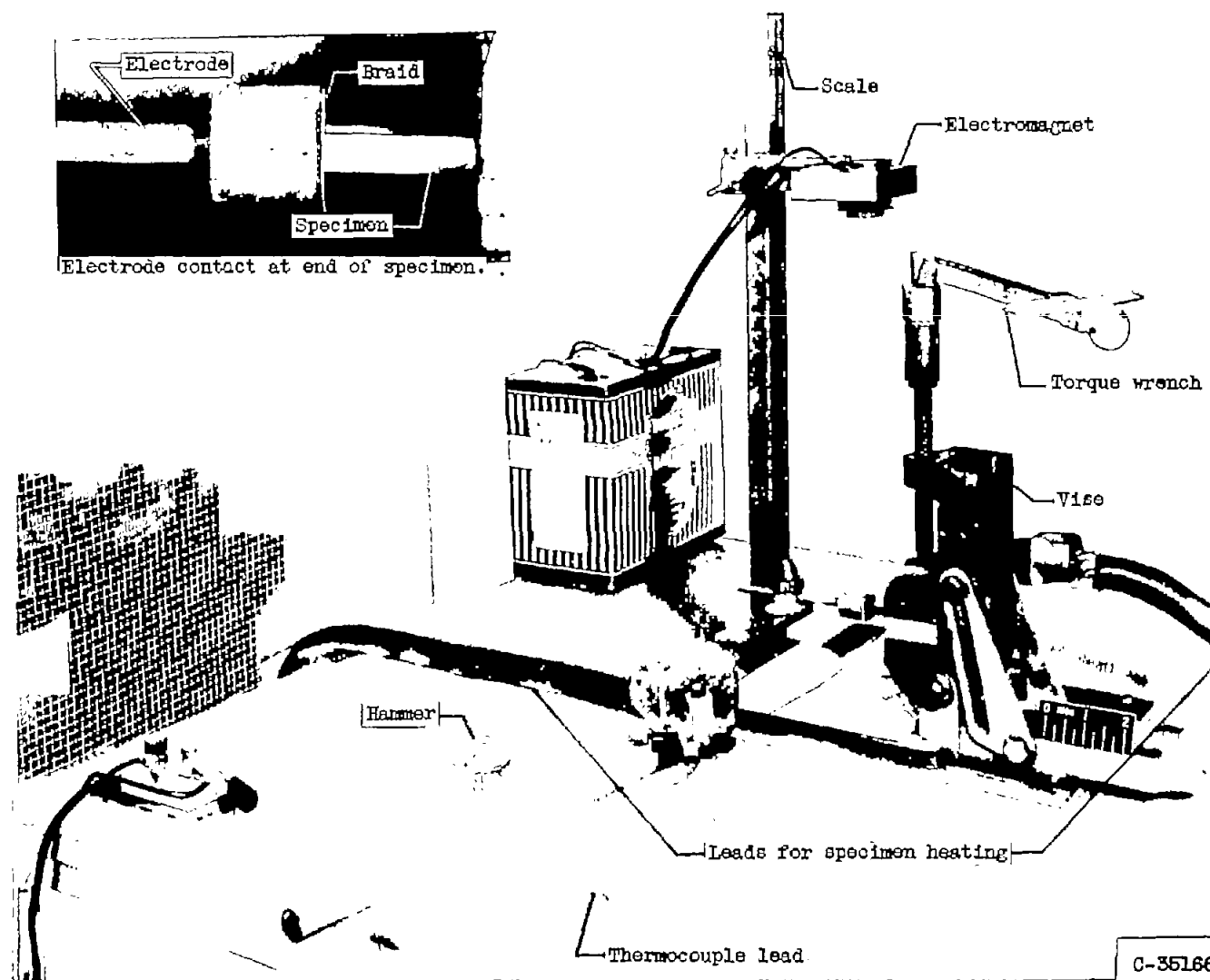


Figure 5. - Impact testing apparatus arranged for high-temperature testing.

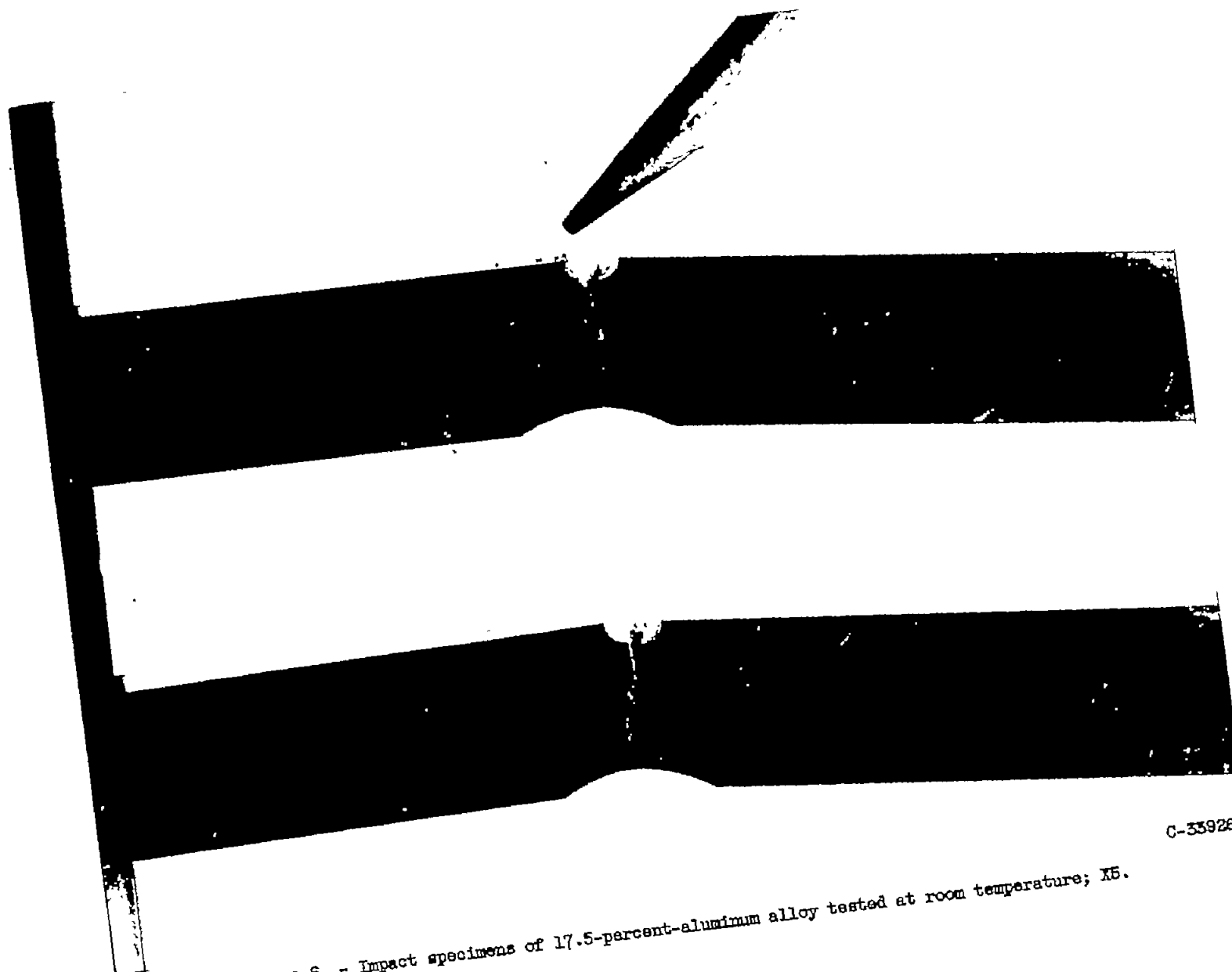


Figure 6. - Impact specimens of 17.5-percent-aluminum alloy tested at room temperature; X5.

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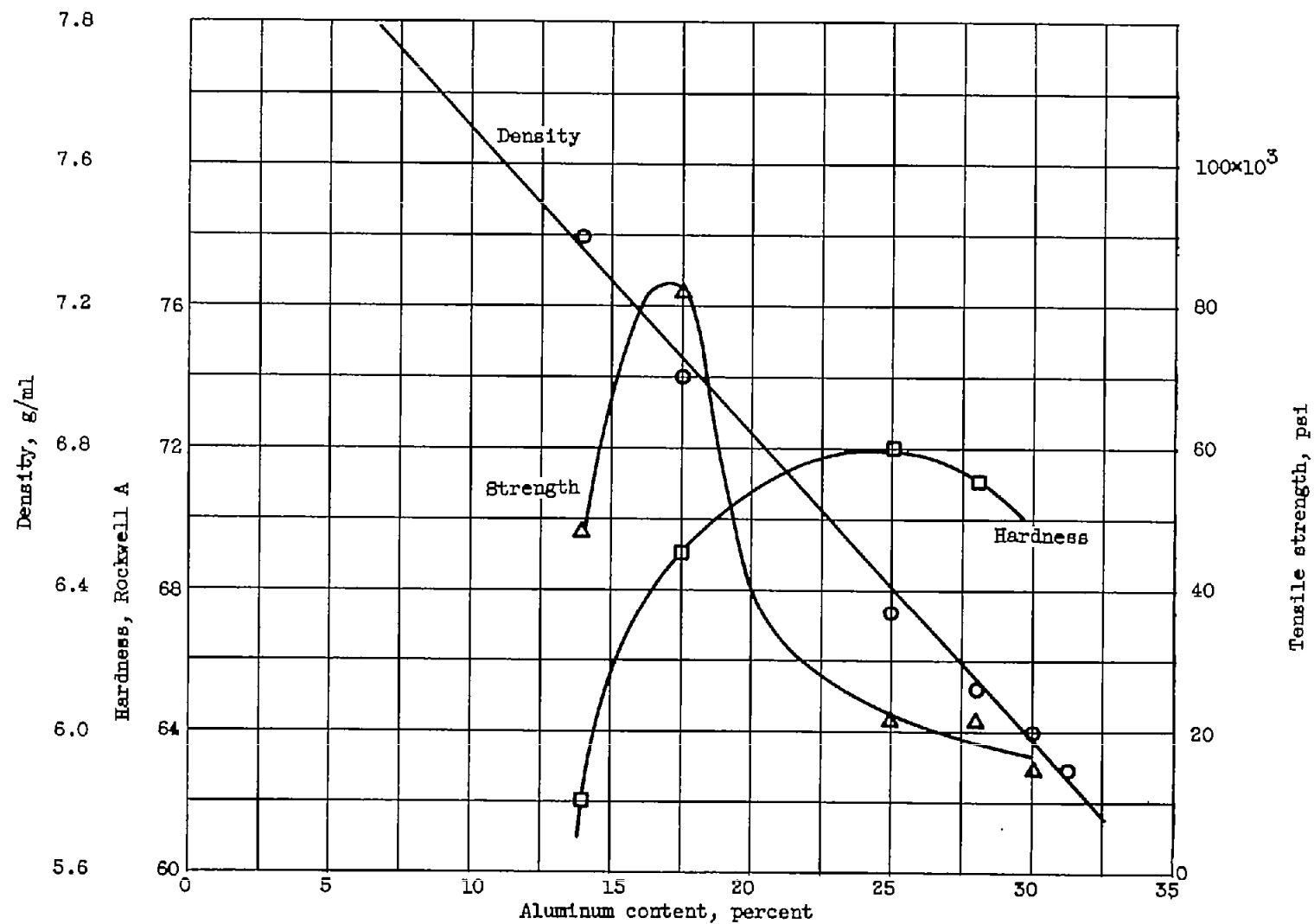


Figure 7. - Effect of aluminum content on density, hardness and strength of nickel-aluminum alloys.

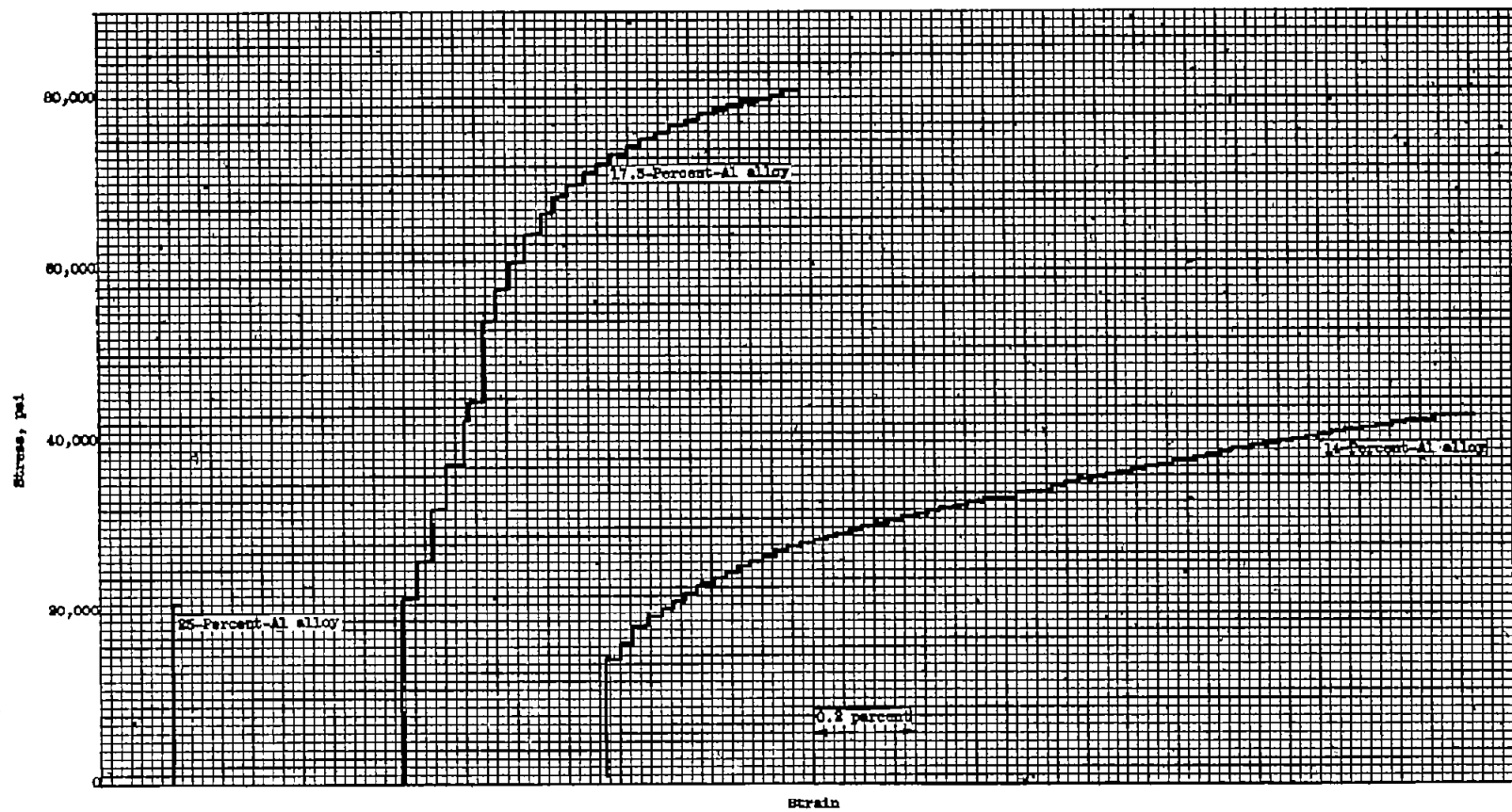
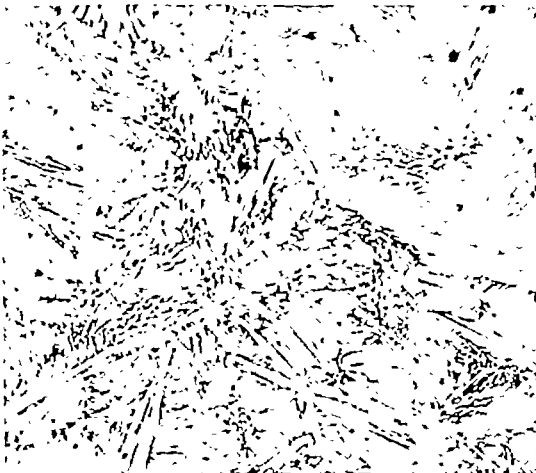


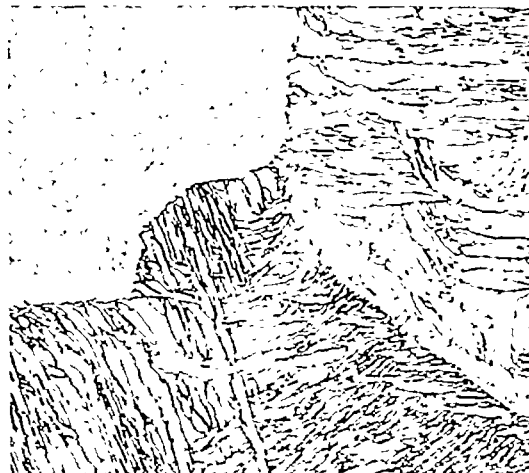
Figure 8. - Representative stress-strain curves for aluminum alloys as taken on separable extensometer.

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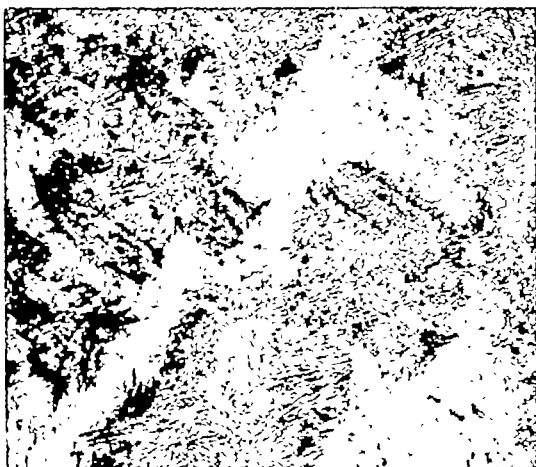
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(a) As-cast structure; X1000.



(b) Rolled 50 percent; air cooled; X1000.

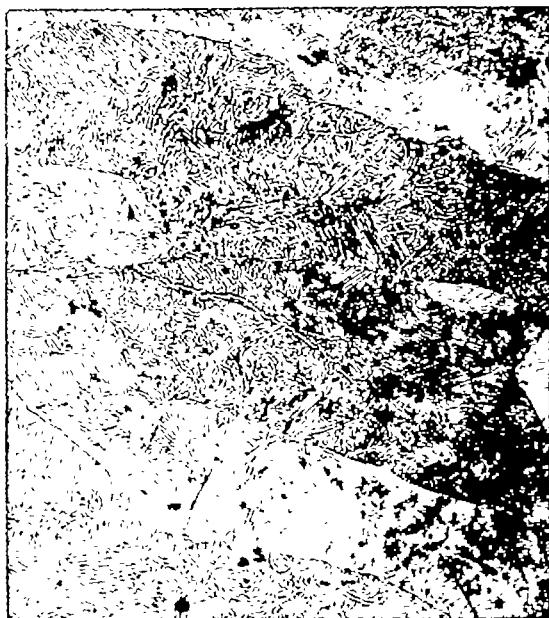


(c) Rolled 50 percent, showing islands of martensite-like NiAl; air quench; X100.

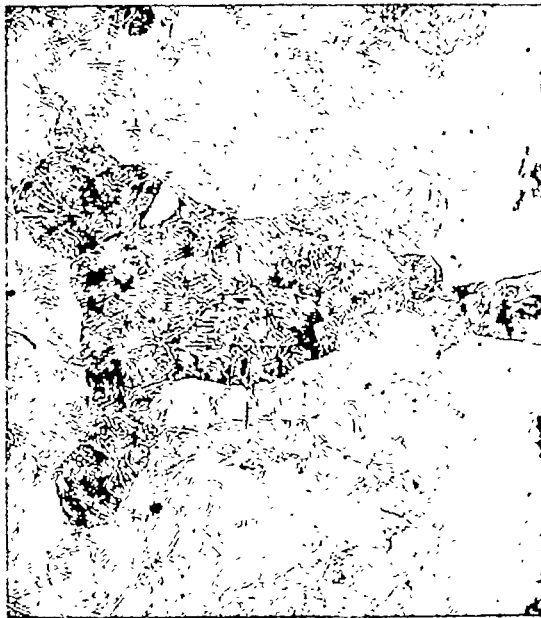


(d) Rolled 50 percent, showing untransformed structure of martensite-like NiAl; air quench; X1000.

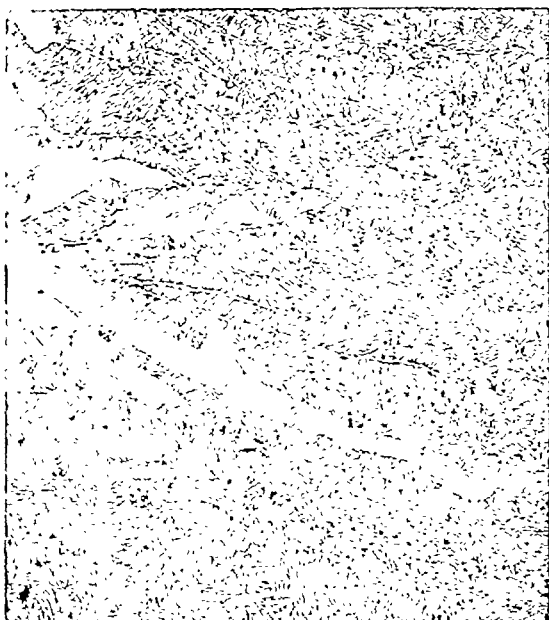
Figure 9. - Effect of rolling at 2400°F on microstructure of 17.5-percent-aluminum alloy. Vilella's reagent.



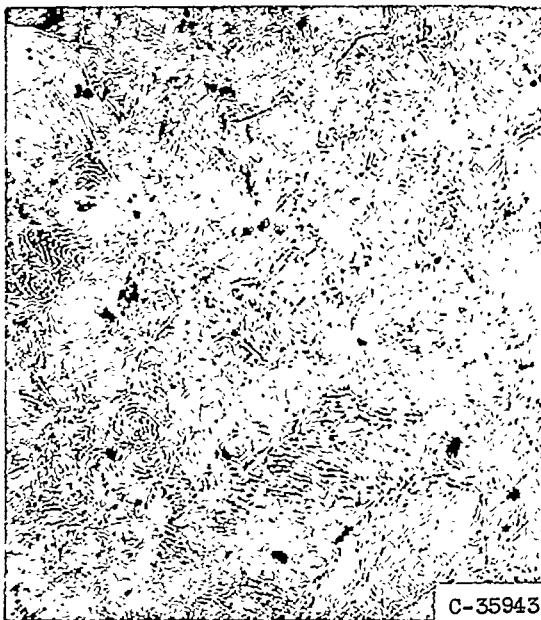
(a) As-cast; no addition.



(b) Titanium added, 0.3 percent.



(c) Boron added, 0.05 percent.



(d) Molybdenum added, 0.5 percent.

Figure 10. - Effect of grain-refining agents on grain size of 17.5-percent-aluminum alloy. Similar areas of castings; Vilella's reagent; X50.

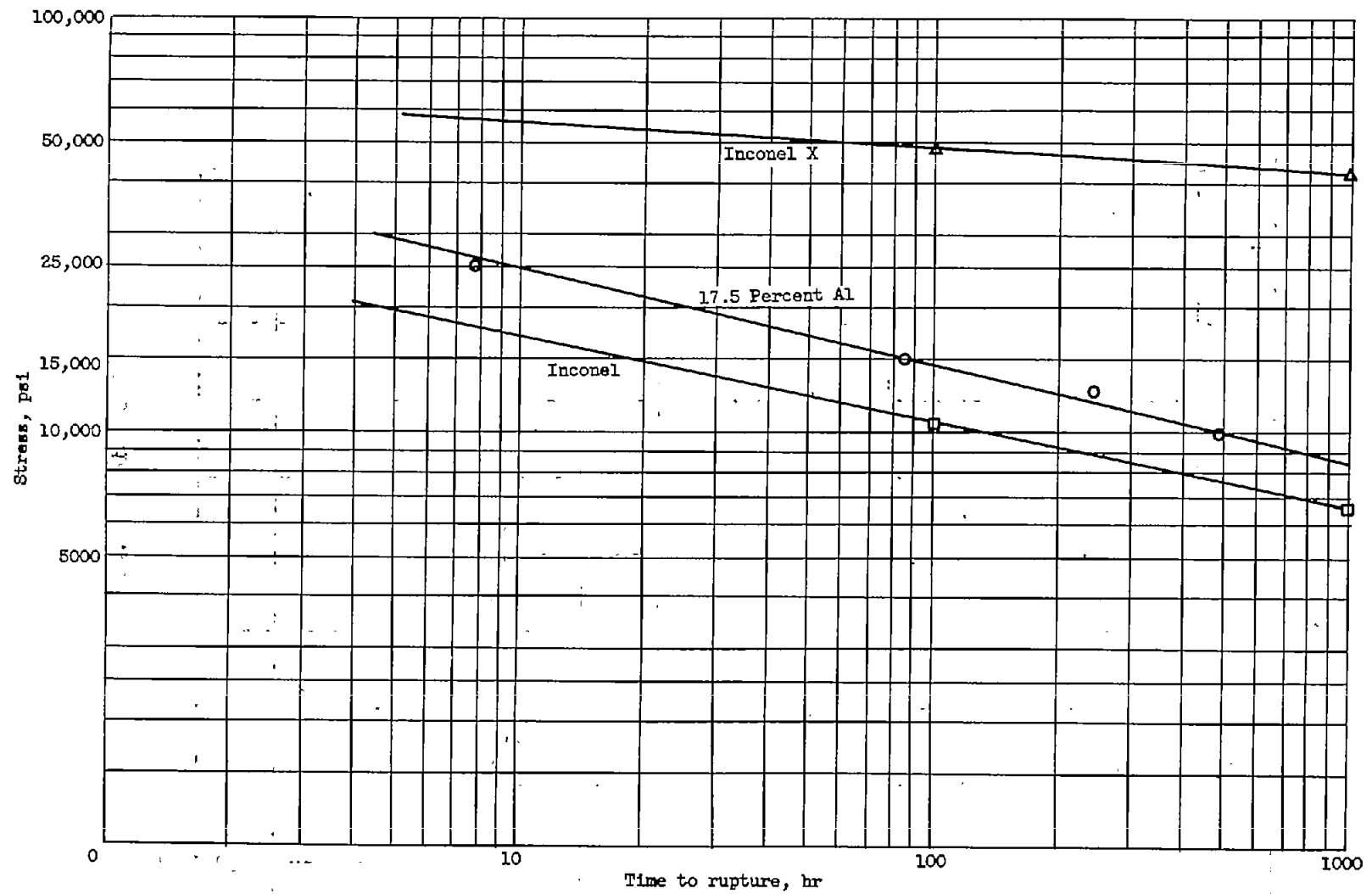


Figure 11. - Stress-rupture properties of 17.5-percent-aluminum alloy at 1350° F.

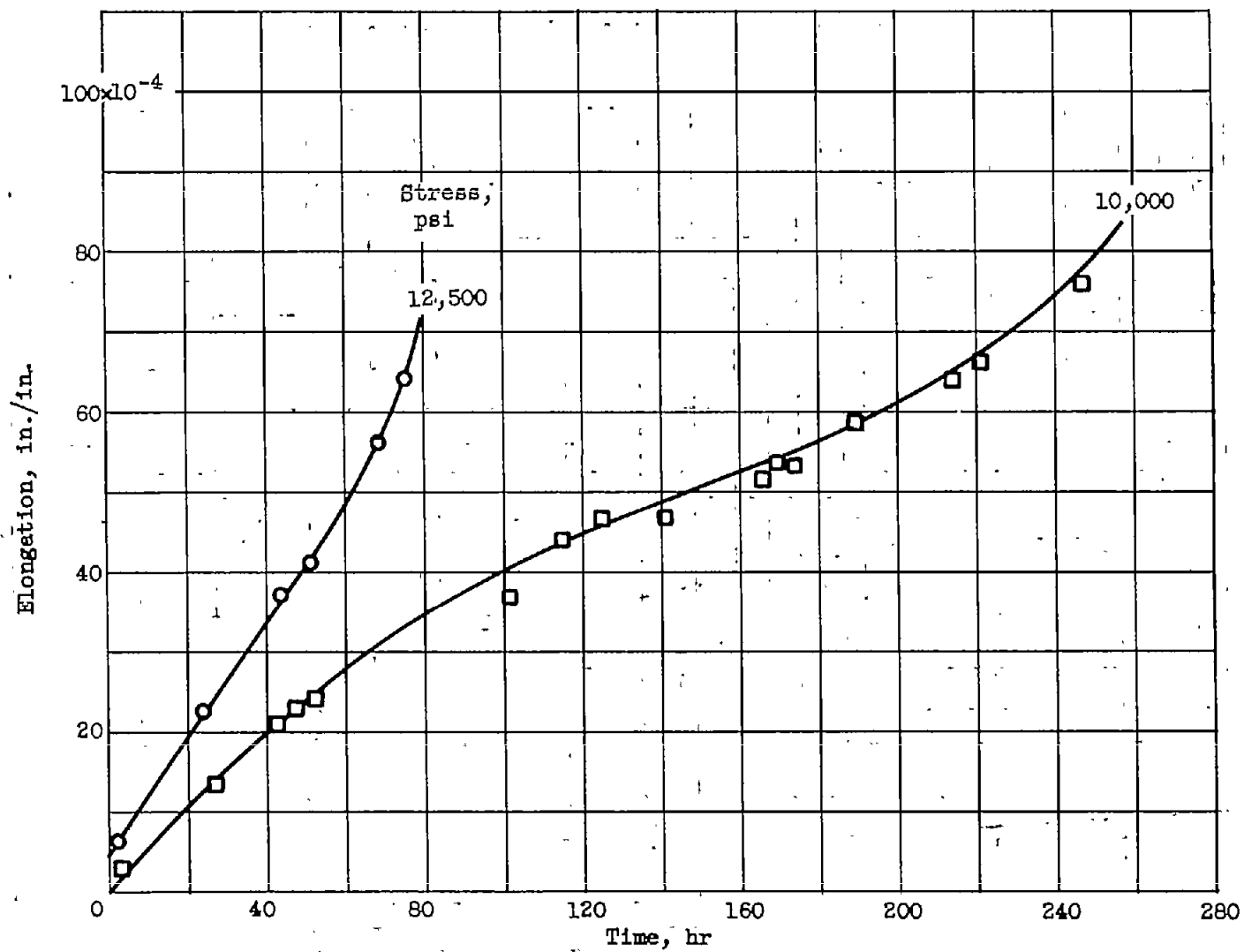


Figure 12. - Creep behavior of 17.5-percent-aluminum alloy at 1350° F.

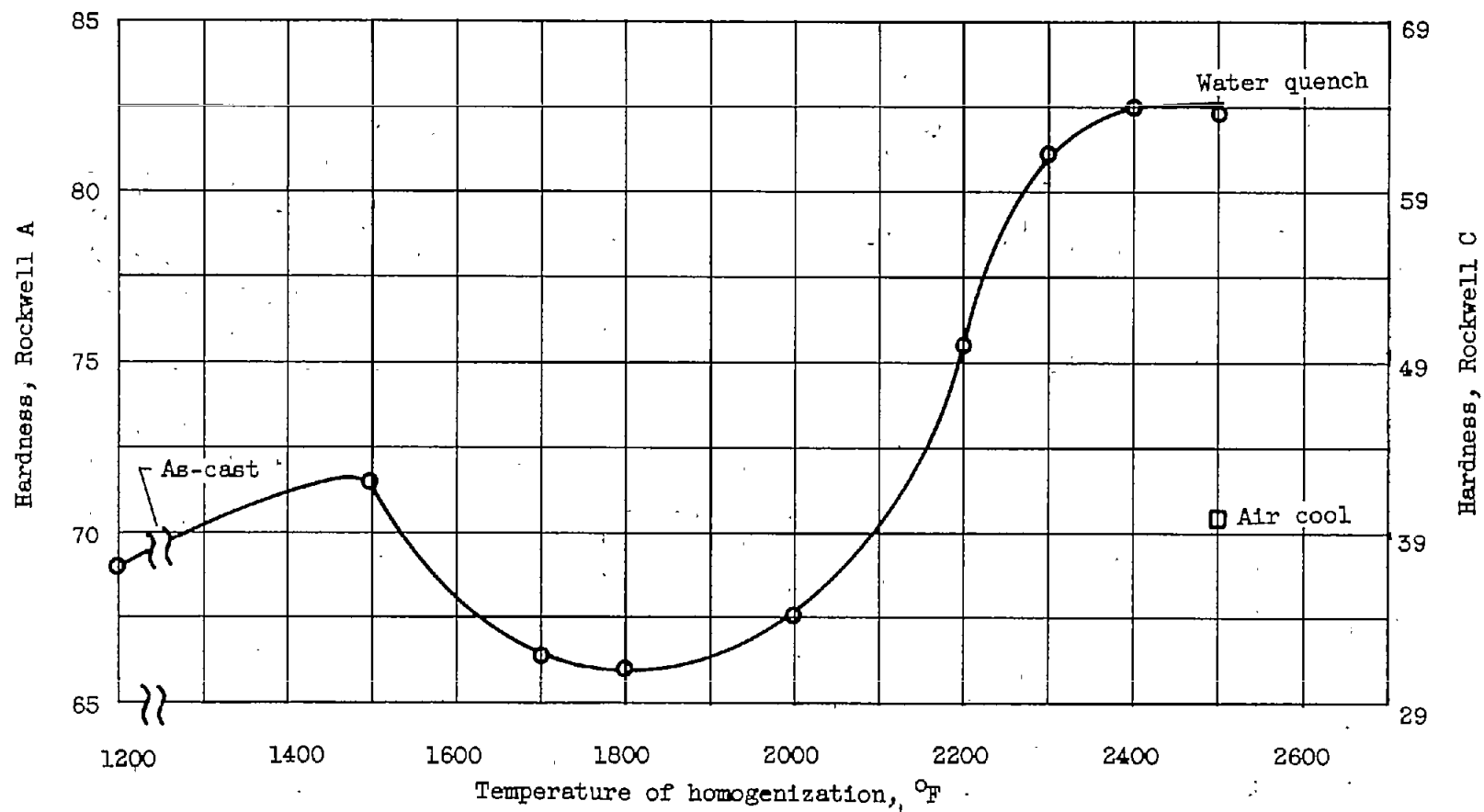


Figure 13. - Effect of homogenization treatments on hardness of 17.5-percent-aluminum alloy.

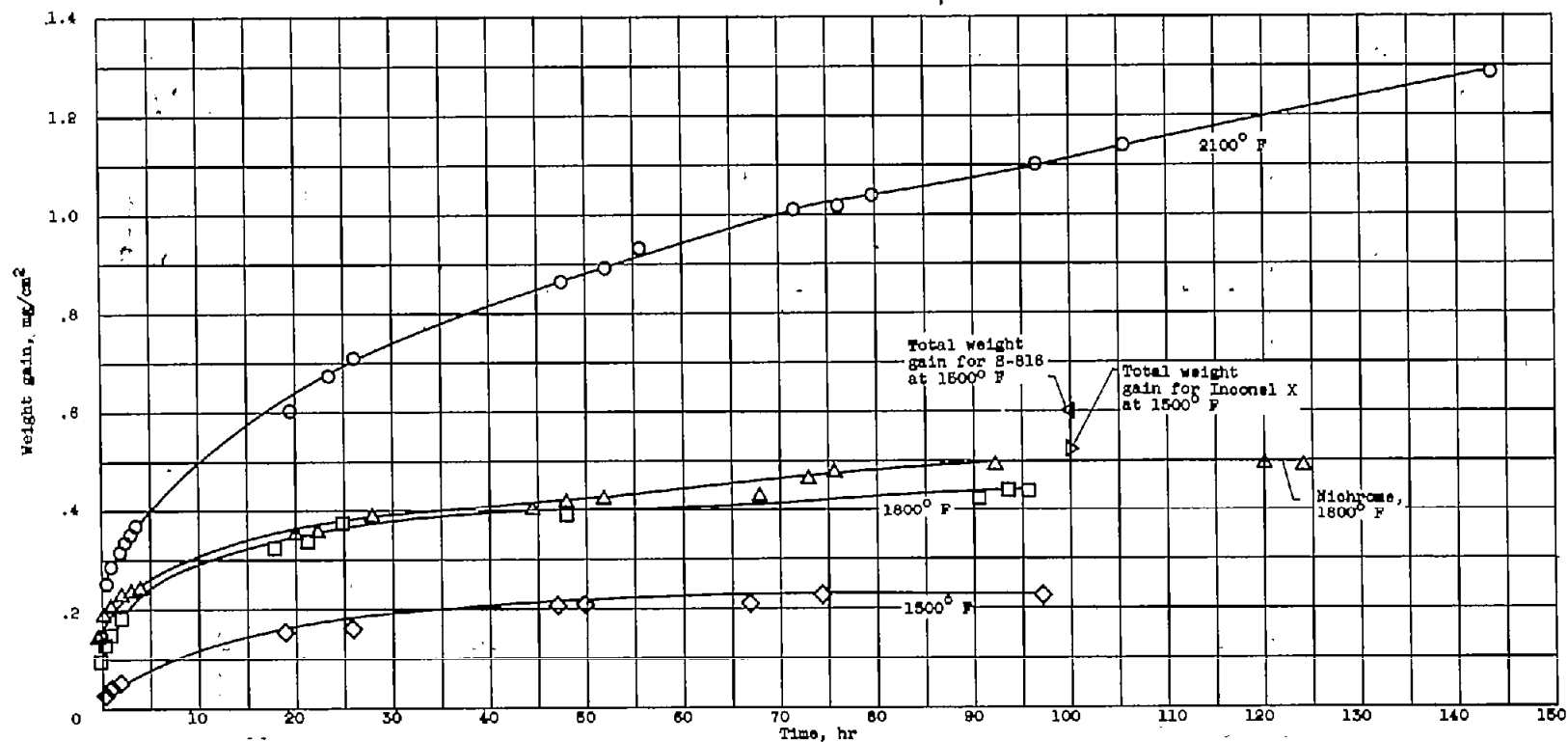
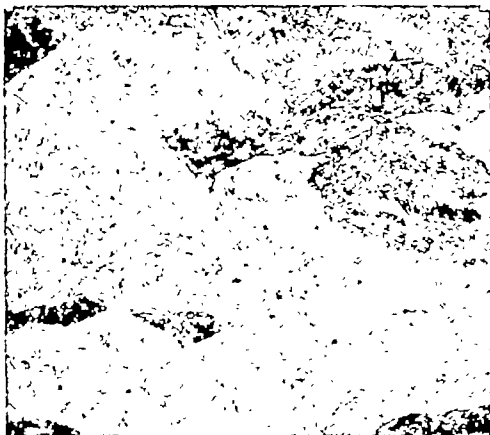
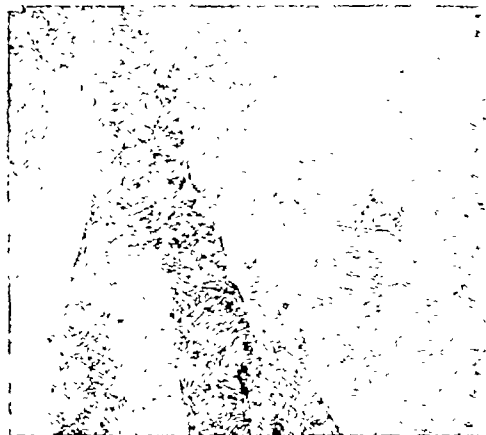


Figure 14. - Oxidation resistance of 17.5-percent-aluminum alloy.

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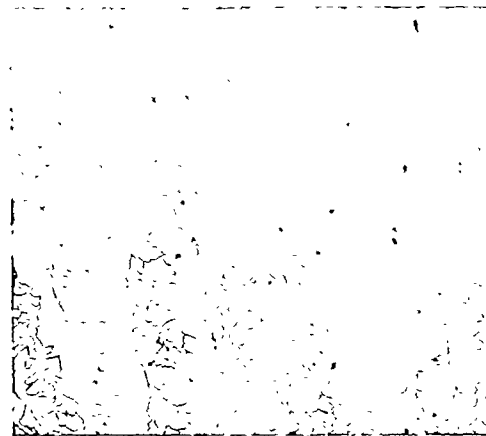
(a) As-cast.



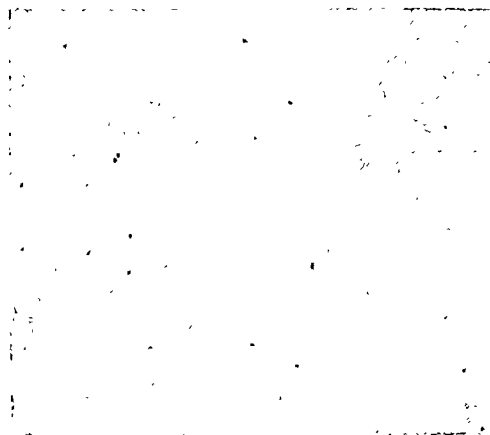
(b) 1500°F, water quench.



(c) 1700°F, water quench.



(d) 1800°F, water quench.

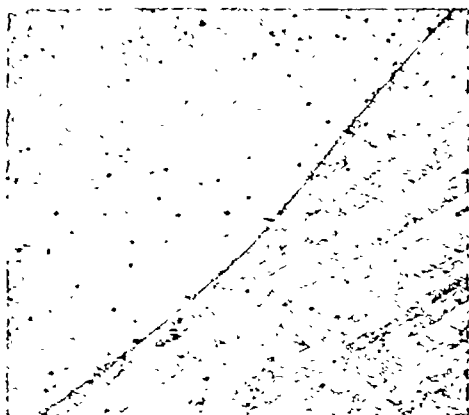


(e) 2000°F, water quench.



(f) 2200°F, water quench.

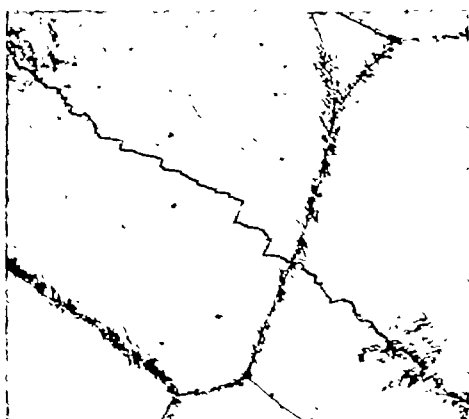
Figure 15. - Effects of 48-hour homogenization treatments at various temperatures on microstructure of 17.5-percent-aluminum alloy. Vilella's reagent; X100.



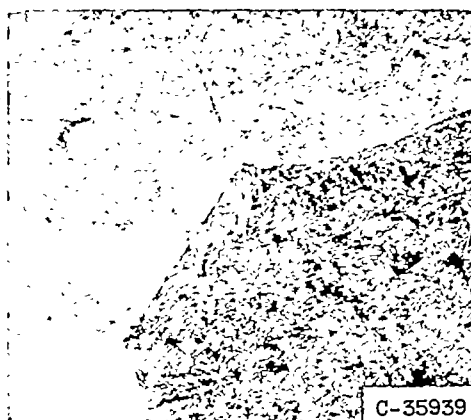
(g) 2300°F; water quench.



(h) 2400°F; water quench.

(i) 2500°F; water quench.
Note quench crack.

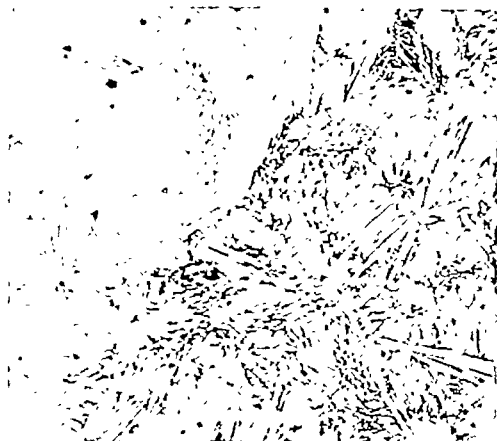
(j) 2500°F; air blast.



(k) 2500°F; air cool.

Figure 15. - Concluded. Effects of 48-hour homogenization treatments at various temperatures on microstructure of 17.5-percent-aluminum alloy. Vilella's reagent; X100.

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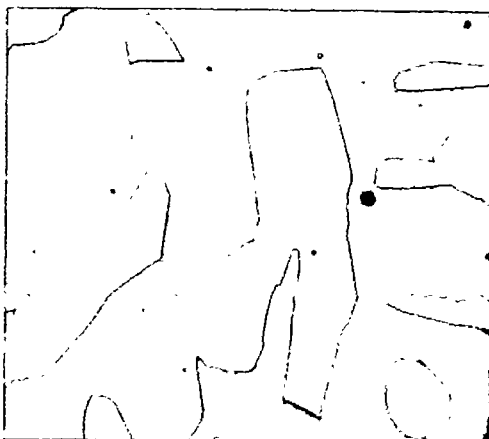
(a) As-cast.



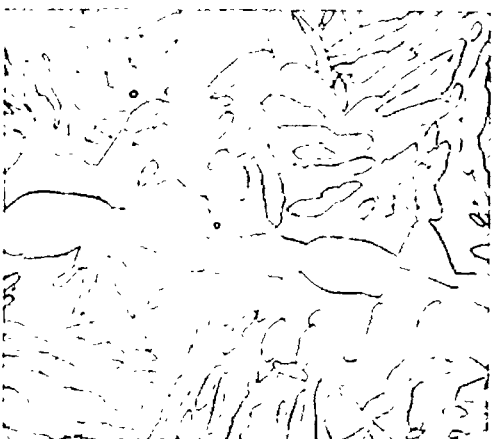
(b) 1500°F, water quench.



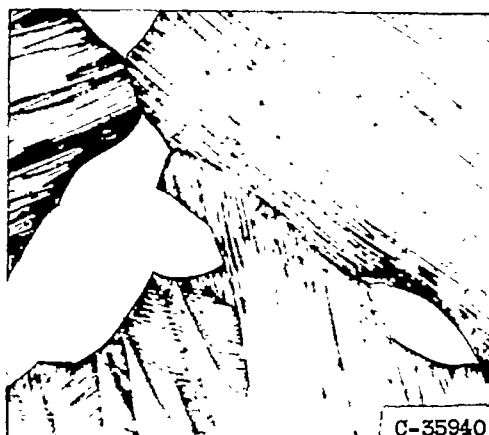
(c) 1700°F, water quench.



(d) 1800°F, water quench.



(e) 2000°F, water quench.

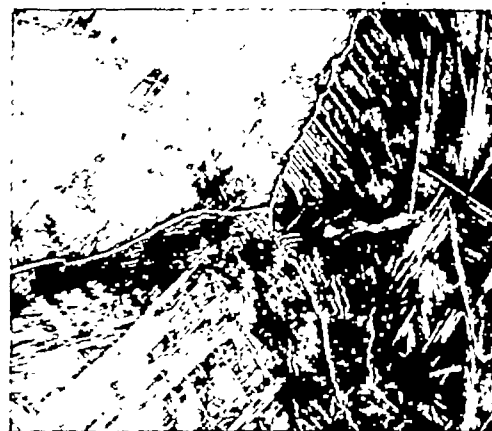


(f) 2200°F, water quench.

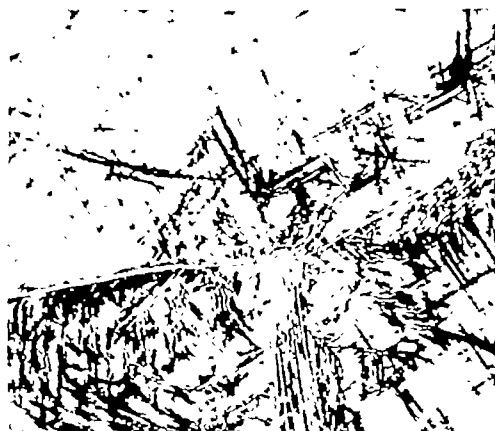
Figure 16. - Effects of 48-hour homogenization treatments at various temperatures on microstructure of 17.5-percent-aluminum alloy. Vilella's reagent; X1000.



(g) 2300°F; water quench.



(h) 2400°F; water quench.



(i) 2500°F; water quench.



(j) 2500°F; air blast.



(k) 2500°F; air cooled.

Figure 16. - Concluded. Effects of 48-hour homogenization treatments at various temperatures on microstructure of 17.5-percent-aluminum alloy. Vilella's reagent; X1000.